AMERICAN SOCIETY
FOR
TESTING MATERIALS

PROCEEDINGS
OF THE
THIRTY-SEVENTH ANNUAL MEETING

Held at Atlantic City, N. J.
June 25-29, 1934

VOLUME 34
PART II. TECHNICAL PAPERS

Published by
AMERICAN SOCIETY FOR TESTING MATERIALS
260 S. Broad Street, Philadelphia, Pa.

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American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART II

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

SUMMARY OF PROCEEDINGS OF THE WASHINGTON REGIONAL MEETING—SYMPOSIUM ON THE OUTDOOR WEATHERING OF METALS AND METALLIC COATINGS

The fifth regional meeting of the Society was held in Washington, D. C., at the Wardman Park Hotel on Wednesday, March 7, 1934, in conjunction with the Spring Group Meeting of A.S.T.M. Committees. Arrangements for the regional meeting were under the auspices of the Washington Committee on Arrangements consisting of:

A. C. Fieldner (*Chairman*), Chief Engineer, Experiment Stations Division, U. S. Bureau of Mines
H. F. Clemmer, Engineer of Tests, Department of Highways, District of Columbia
W. M. Corse, Metallurgical and Chemical Engineer
F. H. Jackson, Senior Engineer of Tests, U. S. Bureau of Public Roads
H. S. Rawdon, Chief, Division of Metallurgy, U. S. Bureau of Standards
Stanton Walker, Director, Engineering and Research Division, National Sand and Gravel Assn.

The afternoon session of the meeting comprised a technical program, a Symposium on the Outdoor Weathering of Metals and Metallic Coatings. There were approximately 250 members and guests in attendance at the session.

The evening session, the Regional Meeting Dinner, was of an informal nature with the following speakers:

Capt. C. A. Jones, Head of the Design Division, Bureau of Engineering, Navy Department
Dr. Robert A. Brady, Special Adviser on Consumers' Standards, N.R.A., and Assistant Professor of Economics, University of California
Col. Henry M. Waite, Deputy Administrator, Federal Emergency Administration of Public Works

Mr. Willard T. Chevalier, Vice-President, McGraw-Hill Publishing Co., acted as toastmaster.

The Symposium on the Outdoor Weathering of Metals and Metallic Coatings was sponsored by the Society's Committee A-5 on Corrosion of Iron and Steel and Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, which assigned the development of the program to a special committee consisting of the following:

F. F. Farnsworth (*Chairman*), Outside Plant Engineering, Bell Telephone Laboratories, Inc.
W. H. Finkeldey, Metallurgist, Singmaster & Breyer
C. L. Hippensteel, Member of Technical Staff, Bell Telephone Laboratories, Inc.
R. F. Passano, Research Engineer, Research Laboratories, The American Rolling Mill Co.
H. S. Rawdon, Chief, Division of Metallurgy, National Bureau of Standards
Sam Tour, Vice-President, Lucius Pitkin, Inc.

The papers of the symposium deal primarily with an analysis of performance data obtained from the outdoor weathering tests carried out by the Society's Committees A-5 and B-3, and from independently made investigations in which the authors have considerable confidence. They illustrate certain proper uses to which Society test data may be put by any materials engineer to solve his daily problems. The authors were not limited to the conclusions or opinions that have been stated officially by the two corrosion committees of the Society. The symposium consisted of the following five papers:

"Outdoor Test Results on Bare and Metal-Coated Ferrous Specimens," by C. D. Hocker, Chemical Engineer, Bell Telephone Laboratories, Inc.
"The Harmony of Outdoor Weathering Tests," by R. F. Passano, Research Engineer, Research Laboratories, The American Rolling Mill Co.
"The Influence of Rainfall and Smoke on the Corrosion of Iron and Steel," by G. N. Schramm, Corrosion Research Laboratory, American Sheet and Tin Plate Company, and E. S. Taylerson, Manager, Research Laboratory, American Sheet and Tin Plate Co.
"The Early Interpretation of Test Results in the Atmospheric Corrosion of Non-Ferrous Metals and Alloys," by W. H. Finkeldey, Metallurgist, Singmaster & Breyer
"Galvanic Corrosion by Contact of Dissimilar Metals," by C. L. Hippensteel, Member of Technical Staff, Bell Telephone Laboratories, Inc.

The chairman of the session was Mr. R. J. McKay, Superintendent, Technical Service, International Nickel Co., with Mr. F. F. Farnsworth acting as co-chairman. The papers elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on the Outdoor Weathering of Metals and Metallic Coatings."

WATER AS AN ENGINEERING AND INDUSTRIAL MATERIAL

EDGAR MARBURG LECTURE[1]

BY SHEPPARD T. POWELL[2]

To be awarded the coveted honor as the choice of this Society to deliver the Ninth Edgar Marburg Lecture is indeed gratifying. It imposes, however, a trust of responsibility which may not be carried lightly, when one considers the heritage upon which the Memorial Lecture was founded. In our feeble effort to honor the memory of Edgar Marburg, who devoted much of his lifetime to the advancement of knowledge of engineering materials, certain phases of the development in this field are herein presented.

The keystone of successful engineering performance is the availability of satisfactory materials. Through the ages, the romantic fulfillment of engineering dreams has been largely a struggle for such materials and has always constituted problems of major importance. The evolution of modern engineering practice is not merely the product of individual effort, but the contributions of many groups, requiring coordination of data culled from research and development in widely diversified fields.

The essential factors in obtaining proper engineering materials for any purpose are a compilation of specifications satisfactory for the specific conditions encountered and adequate structural and testing procedure to produce and evaluate the desired product. Obviously, it would be superfluous to discuss such requirements, since they are the motivating force for the Society's activity. It is my purpose merely to draw attention to them with specific reference to water as an engineering material, and to discuss existing trends in this field of applied chemistry.

Whether or not water may be classified as an engineering material depends on the accepted definition of such a product. If we include any material essential for the completion of an engineering program or required for continued industrial progress, then water automatically comes within such classification. Few engineering or industrial projects may be performed successfully without an adequate and properly conditioned water supply. As with steel or other materials required for a variety of conditions, no general specification for water may be prepared. Water of given quality, satisfactory for one set of conditions, may be totally unfit for another purpose.

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[1] Read on June 27, 1934, before the Annual Meeting of the American Society for Testing Materials Atlantic City, N. J.

[2] Consulting Chemical Engineer, Baltimore, Md.

Granting for the moment that water is a basic engineering material, are there available sufficient fundamental data to assure uniform products applicable for all industrial requirements? Where broad specifications are acceptable, as in the majority of usages, no particular difficulty may be experienced. For critical conditions, however, such data are lacking, and our knowledge on many points is still very much in the twilight zone. Such a viewpoint is obvious when we pause to consider that we are dealing, not merely with a simple compound of hydrogen and oxygen, but complex chemical solutions, the composition of which is seldom accurately known.

There is a natural tendency to ignore the presence of minute quantities of metals in water as insignificant factors. It is doubtful whether such an attitude is justifiable, since up to the present, the data available have been too meager to determine intelligently their industrial or sanitary significance. It is conceivable that even traces of the heavy metals may markedly influence the adequate water processing required for the finer arts. Manganese, for instance, when present in water in amounts as small as one part in three million parts of water is highly objectionable in the dyeing, textile, leather, laundry and soft-drink industries. Further, relatively minor changes in the hydrogen ion concentration of industrial water supplies has resulted in heavy spoilage of material in tin-weighting processes as required in the silk industry.

Experience has fairly well established definite specifications for water requirements in most industries and has indicated tolerances beyond which satisfactory performance is not obtainable. Under critical operating conditions, therefore, accurate testing methods must be used. Routine analytical procedure as used for general water testing is wholly inadequate for many conditions. For example, condensate from high-pressure steam lines may contain only a few milligrams of solids per liter, yet under certain conditions will result in a severe operating problem. The most expert technicians frequently encounter difficulty in determining even the common constituents in water, due to the inherent limitations of quantitative analytical methods. Under ordinary conditions a high degree of analytical precision is not imperative, but as improvement in the art develops, greater accuracy becomes essential. The solution of many problems in water conditioning would be readily indicated if the contributing influence of minor amounts of constituents were definitely known.

Recent development in analytical procedure by micro-analysis, by spectrographic and by electrical methods, forecast the solution of many problems now insolvable.

The recent trends in all forms of industrial development and expansion have demonstrated the necessity for constructive research in this field. Water purification satisfactory for municipal uses is frequently wholly unsuited for many industrial processes. It is obviously impracticable so to condition the public supply of an entire community and have the resulting product conform to the specific requirements of groups of industries. All that industries depending upon public water supplies may reasonably expect is that the raw product delivered shall fall within certain broad specifications. Refined processing for special usage is a function of private management. Under conditions where industries depend on private water sources analogous treatment will occur, the only difference being the relative quality of the raw material. Beyond this point the refinement of the product will be dependent upon specific requirements.

REMOVAL OF SUSPENDED SOLIDS FROM WATER

Practically all surface waters contain some suspended matter which requires removal before the waters are suitable for industrial uses. Even where water is used merely for cooling, removal of gross amounts of suspended solids is necessary and some form of treatment must be employed. The simplest method of clarification is by plain sedimentation. The relative simplicity of such treatment has always attracted attention and the conditions which influence this phenomenon have been subject to critical studies. Scientifically, the problem of water clarification is somewhat involved and the influence of the many contributing factors is not always apparent. The fundamental hydraulic principles involved, occurring when solids deposit from water, are most accurately expressed by the résumé of Hazen(1),[1] and although they were advanced by him more than thirty years ago, they are still generally accepted:

"The fundamental proposition, in clearing water by sedimentation, seems to be that every particle of sediment moves downward through the water at a velocity depending upon its size and weight and upon the viscosity of the water. Particles of sediment are generally so far apart that they do not influence each other; and, while there is no doubt that they do sometimes collect in groups and thus change the conditions, it seems to be generally true that each particle will settle as if no other particle were present.

---

[1] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 45.

"If the water in a basin were absolutely quiet there would be a regular sequence of clearing beginning at the top. The coarsest particles would go down fastest, but at any given point there would be a gradual clearing, and this clearing would take place most rapidly at the top, and, after longer intervals, at lower points in the basin.

"Seddon started out with this theory, but found it to be not in accordance with the facts. His observation showed that while the amount of sediment in the water in the top was a little less than in the water in the bottom, the distribution was nearly equal throughout the mass, a condition of affairs inconsistent with the theory. He accounted for this distribution of sediment by the constant mixing of the water from top to bottom, and to the sustaining power of vortex motions in the water. These motions, he thought, arose from the internal motion of the water at the time of entrance, and from wind, and from temperature changes.

"The writer has taken Seddon's development of the case as his starting point, and has carried the discussion further. He believes that while the internal motions keep the water mixed, and with nearly the same density of sediment from top to bottom, the tendency of the particles of sediment to settle is nevertheless an unbalanced force always acting to take the particles to the bottom, and the number of particles that hit the bottom in a given time is proportional, first, to the velocity at which the individual particles settle, and second, to the density of sediment in the water immediately above the bottom.

"With these fundamental relations in mind, it is easy to compute and to express by simple formulas the proportions of particles of sediment of a given hydraulic value which will hit the bottom under given conditions and which, therefore, presumably will be removed.

"The fundamental propositions may be very concisely expressed. They are: first, that the results obtained are dependent upon the area of bottom surface exposed to receive sediment, and that they are entirely independent of the depth of the basin; and, second, that the best results are obtained when the basins are arranged so that the incoming water containing the maximum quantity of sediment is kept from mixing with water which is partially clarified. In other words, the best results are obtained where any given lot of water goes through the basin with the least mixing with the water which enters after it. This is practically accomplished by dividing the basins into consecutive apartments by baffles or otherwise.

"Thus far, the discussion is easy and apparently certain. The next step is a more difficult one. It relates to bottom velocities, and has to do with the question whether these velocities are such as to allow the particles to remain on the bottom when they get there, or whether they will be taken up again and be kept in motion with the body of the water. This is a point upon which further experimental data are needed. The problem of securing such data seems to be difficult. The observations must be made at the bottom of a layer of liquid of considerable thickness, where the conditions of observation are not favorable. The observations, further, must be made on very low velocities and on particles so small as to be practically microscopic.

"Whatever view may be taken of the second part of the problem, or whatever researches upon it may show, the arrangements of basins most favorable to taking particles to the bottom should stand."

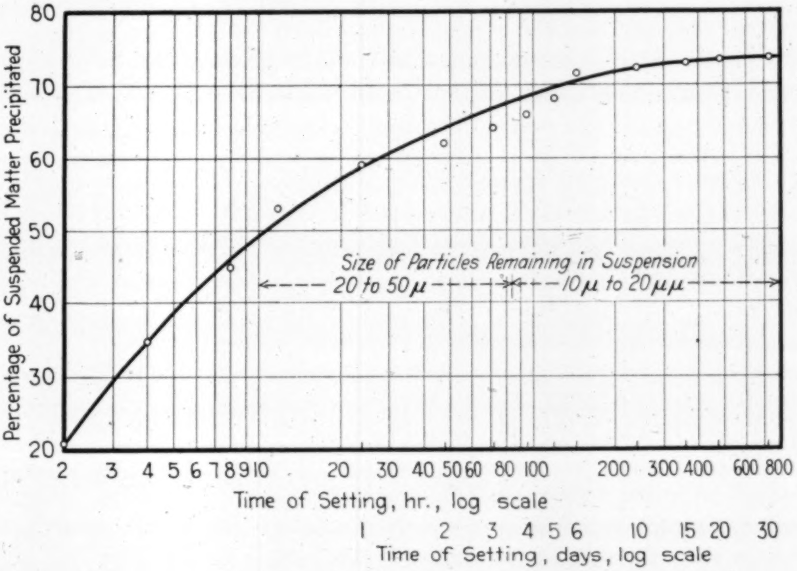

FIG. 1.—Removal of Suspended Solids from Water by Subsidence in Quiescent Water at 62 F. (17 C.).

Even though there may be no horizontal movement of the water, complete clarification is not obtained and long periods of retention of water in a quiescent stage will not completely eliminate finely dispersed solids. This is illustrated in Fig. 1, showing the trend towards equilibrium in respect to classification of particle sizes remaining in suspension. From an engineering viewpoint, there are economic limits beyond which clarification by plain subsidence only becomes impractical. Since the removal of all suspended matter may not be entirely effected by gravity, coagulation and filtration processes have been developed to speed up clarification.

Chemical coagulation has been widely practiced for ages, but the function of the many forces involved in these processes are still vague and imperfectly understood. Hannan(2), in a thoughtful discussion on the subject, has termed these phenomena "micro-forces," and considers the ultimate influence of low potential energy capable only of acting on masses of molecular proportion and limited to a minute range. This investigator has advanced the theory that in liquid-liquid, gas-liquid and liquid-solid interfaces the molecules "marshall themselves in layers, each layer differing probably to some slight extent from its neighbor." Molecular orientation in fluids is thought to be replaced by minute stresses in the case of solids. Summarizing this theory, Hannan believes the whole may be referred to as an interfacial system and that at the intersection of interfacial systems, and more especially at the solid-liquid-gas junction, the micro-forces are most manifested. Continuing further he suggests that water surfaces are oriented molecules of many thousand layers. Pursuing this thought, he believed that "When the water surface takes a curved form, the electromagnetic field thereby developed is such that the following laws obtain: (*a*) convex attracts convex, (*b*) concave attracts concave, and (*c*) convex and concave are mutually repellent. Not only so, but the degree of attraction or repulsion is seen to vary with the curvature. If curvature fineness or division or degree of dispersion are all virtually synonymous terms, we have the stage set for the play of forces in interfacial phenomena that must produce the characteristic results observed in and around the capillaries formed by the deposits of coagulum about the sand grains of a filter bed."

The amount of coagulant required to effect good clarification varies with the nature and amounts of suspended and the soluble solids contained in the water. The work of Theriault and Clark, Miller and others on the formation of floc by alum solution has proved that the acidity of the water, as measured by the hydrogen-ion concentration of the final mixture of alum and water, is of great importance in the production of floc. A portion of the summary of Theriault and Clark's(3) study is given herewith:

"When other possible factors are left out of consideration, the optimum conditions for floc formation will be found within a narrow zone of pH centered for dilute solutions at pH 5.5. The more dilute the water in total salt content and the less alum added, the narrower becomes the pH zone with which optimum floc formation is to be found."

It should be borne in mind that optimum floc formation is not always synonymous with clarification, since satisfactory agglomeration of flocculated coagulants is influenced by a number of factors. Temperature, peptizing agents, certain buffer salts and velocity of mixing greatly affect the results.

As a rule the dose of chemical necessary for proper treatment is proportional to the turbidity of the water. No definite scale of amounts applicable to all waters is possible, since many substances influence materially the coagulating power of water. The quantity of the chemical required to bring about high removal of suspended solids not only changes with different waters but varies from time to time in the same water. Organic matter has a marked effect on coagulation, some organic substances accelerating coagulation while others inhibit this action.

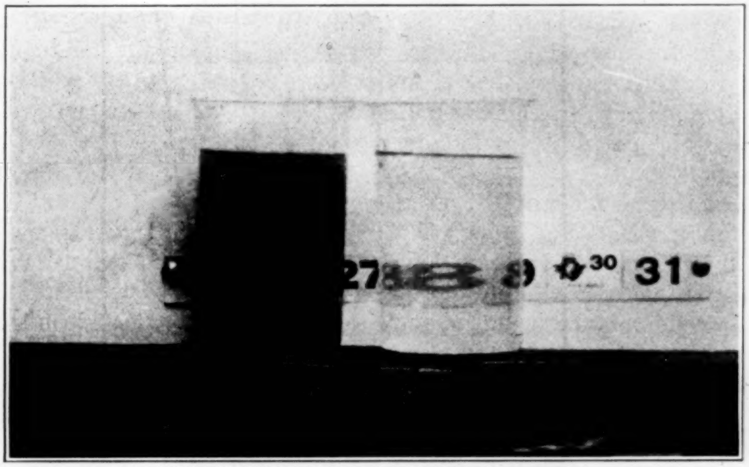

FIG. 2.—Showing Effect of Mixing 15 min.

Beaker on left—Water taken at inlet of sedimentation basin.
Treatment—2.7 grains per gal. alum and 2.1 grains per gal. soda ash, settled 1½ hr.
Beaker on right—Sample of water; same source after 15 min. mechanical mixing, settled 1½ hr.
Turbidity of untreated water 700 p.p.m.

Finely divided mineral matter, such as clays and siliceous compound, has a retarding effect on coagulation. Waters containing large amounts of these constituents require relatively larger doses of chemicals to effect coagulation than would be required for the clarification of waters containing high amounts of readily settleable solids. Many waters in which silica and silicic acid occur as colloids are difficult to coagulate, and purification of such waters presents a difficult problem. Certain organic acids, particularly tannic acid, inhibit the precipitation of aluminum hydroxide, and the failure of aluminum sulfate to coagulate, in many waters, is due to the presence of these compounds.

Smith(4) has shown that "In the coagulation of water containing colloidal clay, the stability of a clay suspension seems to be intimately related to concentration of sodium and potassium carbonate and sulfate. The amount of alum required will be greater the larger the concentration of these, and less as the ratio of calcium plus magnesium to sodium increases."

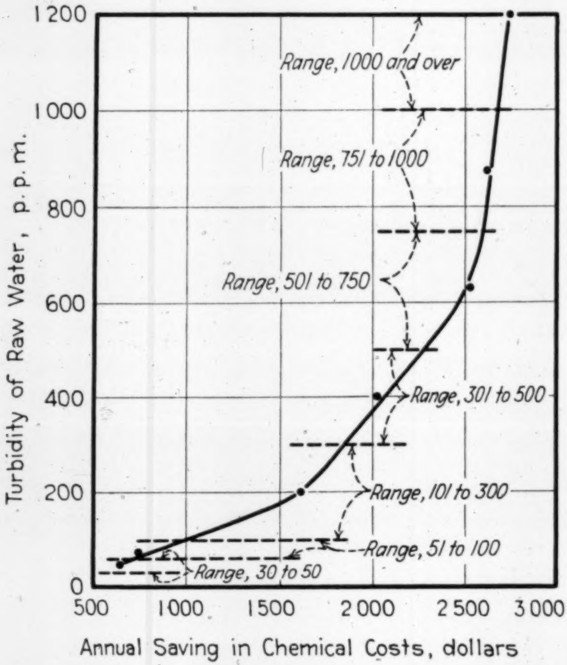

FIG. 3.—Saving in Cost of Chemical Coagulant, per Million Gallons of Water Filtered per Year, Resulting from Substituting 15 min. of Mechanical Mixing of Water and Coagulant in Place of Mixing by Baffles.

The production of clear water, so necessary in many of the processes, involves primarily two specific steps: namely, adequate pretreatment to coagulate suspended or soluble solids, and effective filtration of the agglomerated particles. It is extremely difficult, except at very low rates of filtration, to meet these requirements if either the coagulating or filtration equipment is inadequate. The thoroughness of the preparation of water prior to filtration will vary widely in different localities, owing to the nature of the suspended or soluble solids carried by the supply. In every case, however, there are two fundamental principles which may not be ignored. These are the use of the proper type of chemical coagulant, and proper admixture of the chemicals and water. For many years it was realized that proper mixing of chemicals and water was an important item for satisfactory water purification. The economic importance of this point, however, was not fully realized until quite recently. To accomplish the best result requires proper adjustment of the mixing velocity and contact period, and other structural or operating details. The effect of adequate mixing is illustrated in Fig. 2 which shows the rapidity and effectiveness of clarification of very muddy water, by agglomeration of the finely dispersed particles into readily settleable masses of coagulum. Economically, such control is highly desirable, since speeding up the rate of clarification is reflected in structural and operating costs of any water purification system. A concrete example of this is demonstrated in the net annual saving in chemicals in a large industrial plant shown in Fig. 3.

At the present time a number of research investigations are being conducted concerning the relative value and efficiency of iron and alumina coagulants. These studies have demonstrated that for certain waters ferric salts, particularly the sulfate, possess specific advantages over many other coagulants and are preferable for the clarification. The ferric salts are especially useful for the clarification of waters containing low alkalinity. Ferric sulfate is particularly adaptable for the treatment of some waters since it has a wide pH coagulating range requiring less careful adjustment of the isoelectric point. The coagulum formed by iron salts is bulky and possesses high absorptive properties.

EFFECT OF COLOR ON INDUSTRIAL WATER SUPPLIES

In certain industries, the colors and shades of manufactured materials require complete removal of all colors from waters used for processing and require the elimination of any organic or inorganic materials which may result in off-color or stained products. These requirements have necessitated effective decolorization methods. True color is due largely to the solution or colloidal suspension of organic material, and waters so stained reflect the organic nature of the watershed from which the water is collected. At times, color may be imparted to the water by certain inorganic substances, but in general organic material is responsible for the staining of both surface and underground supplies. The discussion of colored waters and methods for their correction is limited here to compounds present in water whether in true solution or in a colloidal state, which are not to be confused with apparent color imparted to the water from suspended solids. In certain sections of the country, especially in many New England and Southern states, highly colored waters are fairly common. In some cases marked reduction in color of water can be effected by holding the supply in impounding reservoirs for long periods. Where a high degree of removal is not desired, this procedure is relatively satisfactory but complete decolorization may not be obtained. Removal of color in this way is the result of the combined action of sunlight and biochemical processes. In some instances high color removal has been effected by filtration of the impounded reservoir water through sand without the aid of coagulants. The exact phenomenon of decolorization of water by impounding is due, probably, to a number of factors. The theory advanced by Hazen and confirmed by others is that during the putrefactive action which occurs in the bottom of reservoirs, the dissolved oxygen in the lower strata of water is exhausted, and the accumulated iron in the reservoir bottom is reduced to a ferrous state. With seasonal changes, the lower layers of water are brought to the top, where aeration takes place. By their contact with oxygen, the ferrous salts are oxidized to a ferric condition and are thus precipitated. Thus, much of the color is adsorbed and precipitated. There are three possible conditions which may result from the biological action followed by aeration. These depend upon the relative amount of iron, the nature of the color, and biological activity. All colored waters are not decolorized by detention in reservoirs, since the combination of iron and organic matter may be so stable as to inhibit removal either by coagulation or filtration.

Accurate data relating to decolorizing water chemically have been secured through a number of investigations. The principle of decolorization by chemical processes is largely one of determining the isoelectric point required for the agglomeration and precipitation of the organic or inorganic compounds responsible for the staining of the supply. The classical studies of Wolman and Hannan (5) in 1921 did much to advance the art of coagulation of substances in waters by chemicals and has considerable bearing on decolorization control procedure. These studies firmly established certain fundamental principles of coagulation which have been responsible for later experimentation relating to color removal. It has been shown by cataphoresis that color in water is largely in a collodial state. As pointed out in the discussion of suspended matter, it is now recognized that careful adjustment of the hydrogen-ion concentration of water is a primary and important factor for effective coagulation of colloids. The precipitation of alum first approaches completion near a pH value of 5.4 and at such values where aluminum ions exist as such, color may be readily removed. This was accomplished by Norcom(6) who decolorized the highly colored water of the Cape Fear River by coagulation and clarification at a pH value of about 5.4. The effectiveness of color removal from water by adjustment of the pH values will vary with the nature of the material responsible for the staining. For instance, water containing colors of the humic acid type, such as occur from drainage of swampy or peaty areas, are frequently difficult to decolorize. Miller(7) studied a number of such water supplies and showed that these colors were largely colloidal; and cataphoresis studies indicated that where the coloring matter was stable (not coagulated) it bore a negative charge. Further, that coagulation and removal of color by electrolysis was effected by the cation, and relative effects of different cations were found to follow Schulze's rule of valence.

With possible limitations in applying the fundamental principles suggested, it is concluded by Miller that, using alum as a coagulant, clarification was due to aluminum ions and that "alum floc," effective in clarifying turbid water, had an unimportant rôle in the removal of colors of the humic acid type. It was shown that decolorization could be obtained either below a pH value of 4.6 or between the values of 6.9 and 8.3. The reaction at the low pH values was shown to be between the negative color ions and the positive aluminum ions, while the reaction at higher pH values forms hydrous aluminum oxide which absorbs the color mechanically.

Decolorization of water has also been obtained by prechlorination followed by alum coagulation. This is probably due to oxidation of organic matter which would normally inhibit color adsorption by the flocculated aluminum compounds, and in addition the direct coagulation of certain colloidal materials brought about by chlorination alone. It is probable, also, that prechlorination accelerates color removal by chlorination of ferrous compounds which in turn act as adsorbing colloids. This theory has been substantiated by a number of investigators who have found that under some conditions excellent color removal is effected by the use of chlorinated iron salts.

Quite recently, the use of activated carbon in water purification has proved the effectiveness of such materials for accelerating color removal, and indicates promise of a practical and relatively inexpensive procedure for speeding up the rate of decolorization of certain waters. Since color in water may be due to the presence of numerous compounds, the successful removal of these compounds is not subject to any generalized form of treatment and, in the majority of cases, the design of a treating system for effecting the best results will require special study. This is particularly desirable in attempting to remove the last traces of color as required by a number of industrial processes.

MANGANESE IN INDUSTRIAL WATER SUPPLIES

Detrimental Effect of Manganese:

Possibly there is no constituent in water more detrimental to industrial processes, or responsible for greater economic losses, than manganese. This metal is generally associated with iron but its removal presents a much more difficult problem than the elimination of iron. Losses in textile, laundry and soft-drink industries, resulting from precipitation of manganese, has focused attention on the need for adequate purification of many surface and well-water supplies containing an appreciable amount of this element. Although manganese has been recognized for centuries as an objectionable constituent of water, satisfactory methods for its elimination have been lacking. Pliny mentions manganese for bleaching glass under the name of "magnes" and considered pyrolusite, an oxide of manganese, as a loadstone, or a variety of magnetic iron ore. Towards the end of the eighteenth century, chemists generally believed that this oxide was essentially an ore of iron. J. H. Pott (1740) proved that pyrolusite proper did not contain iron, and produced a number of salts quite different from those obtained with the iron oxides.

Although manganese occurs in water less frequently than does iron, manganese-bearing waters are more abundant than is generally recognized. This is because it is usually overlooked owing to its similarity to iron and is therefore not generally separated in routine chemical analyses. As a rule, manganese is present more often in water from underground sources than in surface water supplies, and usually appears as the result of decomposition of manganiferous deposits by the solvent action of weak acids present in the soil or water. Frequently manganese is taken into solution from deposits in the bottom of reservoirs where stagnation occurs, a condition favoring the production of hydrogen sulfide, carbon dioxide, and other compounds which act as solvents on the manganic ore. Unlike iron, manganese has a multiplicity of valences and is capable of performing the functions of an acid as well as a base. The frequency of manganese-bearing waters in some sections is indicated by a number of recent surveys. The New Jersey Department of Health(8) found that the manganese content in 238 of the potable water supplies of that state ranged from 0.03 to 0.20 p.p.m. In Illinois Corson(9) reported that out of 147 waters from wells, 60 showed a manganese content of from

0.03 to 2.8 p.p.m. Of 10 spring waters, 6 contained no manganese, while 3 contained 0.40 and one 7.8 p.p.m., while 11 of the 16 streams examined contained manganese in amounts of from 0.02 to 0.09 p.p.m.

The most notable occurrence of manganese in water was the so-called "manganese calamity" of Breslau, Germany, in 1906(10). The Oder River rose and overflowed the well field, increasing the iron content to 440 p.p.m. and the manganese content to 220 p.p.m. This load far exceeded the capacity of the iron removal plant, and manganese and iron both passed through the filters, requiring the temporary abandonment of the supply. The generally accepted explanation of this unusual condition was that during the preceding dry period, the manganese in the peaty top soil had dissolved as a sulfate, and the high water which followed readily leached the soluble manganese salt into the water-bearing strata below. Some idea of the relative manganese content of ground and surface waters in this country and a few European cities may be obtained from Table I.

TABLE I.—MANGANESE CONTENTS.[1]

Ground Waters		Surface Waters	
Locality	Manganese, p.p.m.	Locality	Manganese, p.p.m.
Amesbury, Mass	0.30	Amherst, Mass	0.01
Babylon, N. Y	0.07	Amsterdam, Holland	0.30
Barnstable, Mass.[a]	0.03	Baltimore, Md	0.20 or less
Bayshore, N. Y	0.37	Barberton, Ohio	0.90
Billerica, Mass	1.60	Boonton, N. J	0.04
Bjornstop, Sweden	3.4 to 53.4	Boston, Mass. (Metropolitan)[a]	0.01
Breslau, Germany	trace to 220	Branchville, N. J	0.08
Brookline, Mass, (avg.)	0.41	Brockton, Mass.[a]	0.01
Calverton, N. Y	0.30	Burlington, N. J	0.10
Cohasset, Mass	0.15 to 0.40	Butler, N. J	0.04
Halle, Germany	1.50	Columbus, Ohio (Scioto River)	0.22
Kingston, Mass.[a]	0.05	East Liverpool, Ohio (Ohio River)	0.88
McKees Rocks, Pa. (Pittsburgh Suburban Water Co.)	1.0 to 4.0	Jersey City, N. J	0.04
Medway, Mass.[a]	0.05	Marlboro, Mass.[a]	0.04
Middleboro, Mass. (avg.)	0.67	New Bedford, Mass.[a]	0.03
Patchogue, N. Y	0.20	New York City (Croton System)	0.03 to 0.10
Reading, Mass	0.004 to 0.56	Ogdensburg, N. J	0.06
Stettin, Germany	5.22	Oakland and Berkeley, Calif	less than 0.10
Superior, Wis	0.12	Phillipsburg, N. J	0.04
		Rahway, N. J	0.03
		Springfield, Mass.[a]	0.14
		Sussex, N. J	0.08
		Wanaque, N. J. (Wanaque Reservoir)	0.00 to 2.5
		Youngstown, Ohio (Meander Creek)	0.22

[a] Results by courtesy of H. W. Clark, Chief Chemist, Massachusetts Department of Public Health.

The most acute industrial problems experienced from manganese-bearing waters have occurred in laundries, in dyeing and in textile establishments and in the soft-drink industry. At Baltimore(11), serious difficulty developed from bleaching yarn and a similar condition was described by Goldthwaite(12). The manganese "epidemic" in Baltimore in 1924 seriously affected the laundries and caused appreciable losses before the cause of the trouble was identified and controlled. At first the laundry owners believed the trouble was due to iron and cleaned and renewed piping systems in their plants. This conclusion was reached since certain fabrics went through the laundry process with practically no stains, while others stained badly and greasy rust spots developed on much of the laundered material. It is common practice in many laundries to pass fabrics through a bleach at the end of the washing process. White dresses and uniforms stained first around the collars and cuffs where such garments come in contact with the body, but no fabrics were entirely free from staining. When the cause of the difficulty was located, the problem was controlled by avoiding the use of extremely caustic compounds and many completed the wash in a slightly acid solution before bleaching. The majority of the laundries used oxalic or other acids. Clothes washed at home were but little affected since strong caustics and bleaches were not used. The staining of white enameled fixtures was greatest in the kitchen where grease was present. In 1925, a comparable manganese problem developed in New York City. Many laundries suddenly complained of spotting clothes and staining of enameled fixtures. Hale(13) found that the difficulty was due to the presence of manganese to the extent of 0.08 p.p.m. in the water supplied from the Croton reservoir.

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[1] R. S. Weston, "Manganese in Water, its Occurence and Removal," *Journal*, Am. Water Works Assn., Vol. 23, No. 9, pp. 1273, 1374 (1931).

Although the laundry industry has probably experienced the greatest amount of difficulty from the use of waters containing manganese, bleaching and textile mills also have been affected seriously. This is particularly the case in communities supplied with soft water containing small amounts of manganese. Rayon mills have likewise suffered from "manganese plague."

Although various writers mention instances of deposits of manganese noted in water mains before 1896, it was probably not until that year that an analytical determination of manganese in ground water was made(14). This test received little attention until 1906 when the City of Breslau(10) experienced serious trouble with its municipal supply.

Black slimes were reported in the system of Newcastle-on-Tyne, England, in 1880(15); and Raumer(16) records manganese-bearing incrustations in pipes in buildings in Bavaria, and in the wells supplying water for use in breweries in Fürth and in Kulmbach, Germany. The first case in America was reported in 1898 in a well at a textile mill in New England(10).

Removal of Manganese:

At the time of the Breslau experience, there were two distinct schools of thought concerning manganese removal from water; one was based on biochemical and the other on inorganic processes. The inorganic process for the removal of manganese from water by chemicals was based upon the original discoveries of Berzelius(31); Tillmans(14), in 1914, established the value of this method. The biochemical process depends upon the activity of certain types of thread bacteria to eliminate manganous or manganic salts. Molisch(17) first observed manganese on one of these organisms and by 1910 six species had been identified. Later many others identified certain specific organisms of this group(17,18,19,20). These and later studies received impetus through iron accumulations causing difficulties in a number of municipal water supplies in Europe. A scientific controversy centered primarily on the question of whether these bacteria needed iron in body processes or whether iron was merely a waste product, and secondly, whether the organisms benefited from the energy created in the breaking up of the compound attacked.

Jackson(21) studied the precipitates in the water distribution system of Newton, Mass. In these deposits, containing high manganese, he found a bacterium resembling *Crenothrix polyspora Cohn*, but it was broader and had thicker walls. Thinking it a new species he named it *Crenothrix Manganifera Jacksonii.* This bacterium is referred to in numerous water supply publications of recent years. Schorler(17) and Ellis(20), however, later showed that this organism is the same as *Crenothrix polyspora Cohn*, and that it actually prefers manganese bicarbonate and grows larger than when dependent on iron bicarbonate alone.

A fairly large group (22,23,24) of manganese-depositing organisms have been identified and their biological functions in the precipitation of manganese have been classified.

In addition to the removal of manganese by biological processes, a number of purely chemical methods have been developed and used successfully. Manganese has been eliminated from water by the removal of carbon dioxide by chalk(25), followed by filtration through pyrolusite. Carbon dioxide undoubtedly inhibits the removal of manganese from water and prevents precipitation, and certain organic materials also act as inhibitors for the precipitation of the metal. Manganic waters high in CO_2 have been successfully treated by relatively long aeration through coke beds followed by removal of the insoluble manganese salts by subsidence and filtration. It has been demonstrated by a number of investigators that catalysis may play a part in the removal of manganese(26,27,28,29,30,31,32,33,34,35,36,37,38,39). This theory, advanced by Corson(26), is that the lower oxides are formed by the action between manganous compounds and manganese dioxide.

There is indisputable evidence that the precipitation of manganese from waters is greatly influenced by the hydrogen-ion concentration and its control. The author has observed that carbon dioxide, organic matter and manganese hold iron in solution and that by precipitation of the iron salts manganese is likewise precipitated. On the basis of this theory, iron and lime are frequently substituted for other coagulants for the removal of manganese from some waters. It has been demonstrated that deposits of manganese in pipe lines generally contain an appreciable quantity of precipitated ferric salts. This has been reported by Sale(40), who demonstrated that deposits in pipe lines in Pierre, S. Dak., contain approximately 44 per cent of manganese and 8 per cent of iron. The water in the system contained only from 2 to 3 per cent manganese. It is probable that these deposits were not associated with biochemical processes but with changes in pH value, since the heaviest deposits were near the wells and the precipitation occurred rapidly after the water entered the distribution system.

Weston(41) calls attention to the fact that, while hard water high in iron is relatively easily purified, soft waters low in iron and pH value but high in organic matter are difficult to purify. All acids, including carbonic acid, interfere with the precipitation of iron and manganese. Consequently it is necessary to provide for correcting the pH value of waters containing these metals as an initial step in purification. With waters of peculiar organic composition, especially those from beneath alluvial deposits or marshy areas, aeration for removal of CO_2 can be carried so far as to make it impossible to coagulate the oxidized iron before filtration. These phenomena have been noticed at Reading, Mass.; Superior, Wis., and other places.

Not all organic matter interferes with the precipitation of iron and manganese. Weston(42) has found that organic matter from deep wells in geologically old deposits tends to assist in their removal, and in turn the iron causes the precipitation of the organic matter. New organic matter, such as extracts of fallen leaves and grasses, has been found to delay iron precipitation while extracts of lignite and some peats assist.

The recognition of the importance of removing very small quantities of manganese from waters is of relatively recent origin. The pioneers in the development of processes for the removal of both iron and manganese and in studies relating thereto have already been mentioned. Iron removal processes were developed earlier and the number of plants is increasing. A notable deficiency in the knowledge of the manganese content of most water supplies and of the difficulties caused by relatively minute amounts in water is undoubtedly responsible for the scarcity of manganese removal plants.

Sawyer(43), in a discussion of a paper by Frisk(44), concludes that there are two distinct approaches to the problem in the treatment of a water containing iron or manganese, or both, as is usually the case where rapid sand filters are involved. One process is planned to maintain a pH value such that the iron and manganese will remain in solution. The other provides for complete oxidation of the iron and manganese before filtration. The first process is applicable to waters containing only moderate amounts of iron and manganese, having, preferably, a pH value of not more than 7.5 and certainly not above 8.0. This limitation of pH value is sufficient to prevent oxidation of iron and manganese accumulations in the filter sand and in the distribution system.

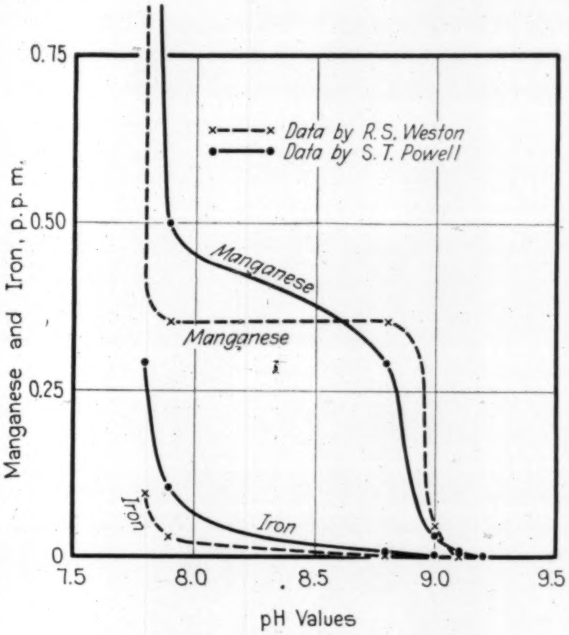

FIG. 4.—Curve Showing Removal of Manganese and Iron at Various pH Values of the Treated Water.

Patrick, at Luke, Md., has been successful in keeping manganese in solution by maintaining a pH value below 8.5. Above this value manganese is precipitated in the mains. The precipitation of manganese varies widely with pH value of the treated water and is influenced by a number of factors. Generally, however, the rate of agglomeration of manganese and removal by subsidence and filtration is effected at higher pH values than is required for the deposition of iron salts. This is illustrated by the curves shown in Fig. 4.

At Providence, R. I.(45), treatment of a soft, colored, impounded surface water with alum was changed in 1931 to treatment with ferrous sulfate and lime. This necessitated cleaning of the filter sand with from 1 to 1.4-per-cent caustic soda solution to remove accumulations of organic matter containing iron and manganese (Fig. 5). Since May, 1932, chlorinated copperas and lime have replaced the ferrous sulfate and lime treatment. For economic reasons, anhydrous ferric sulfate has since replaced the chlorinated copperas treatment. Since iron coagulants have been used at Providence, the sand has remained in excellent condition.

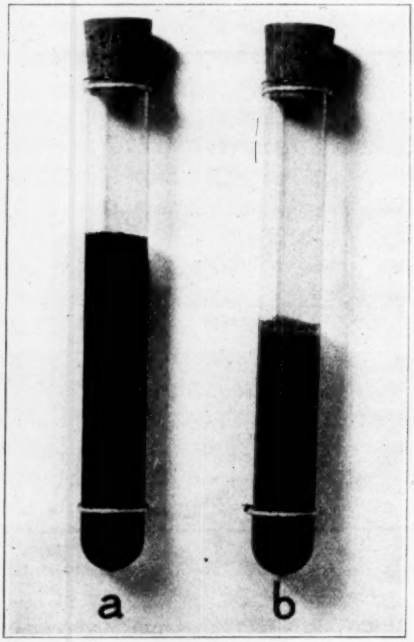

FIG. 5.—Sample of Sand, Showing Reduction in Volume Resulting from Removing Manganese Dioxide Coating.

(*a*) Sand coated with manganese.
(*b*) Same volume of sand after cleaning.

Ferrous sulfate and lime has been used successfully for the removal of manganese at Baltimore, Md., for a number of years. Maintenance of an alkalinity in the mixing basin equivalent to a pH value of 9.0 has been found necessary by Hopkins(46) to produce maximum flocculation and complete manganese removal. Studies with ferrous and ferric salts gave similar results, indicating that the phenomenon is selective surface adsorption by the hydrated ferric oxide floc. Hopkins(47) contends that manganese may be converted to the hydrated oxide by the action of air or dissolved oxygen in the water, and that reaction with alkali produces this compound. Coagulation with iron and lime is believed to proceed at the high pH values in two systems. It is suggested that the manganese is adsorbed by the ferric oxide, but since hydrated manganous oxide is also present, this substance adsorbs colloidal manganese particles, giving a double value to the treatment; and, since the ratio of iron floc to manganese floc is high, most of the work is done by the iron. It is Hopkins' claim that the adsorption of the hydrated manganous oxide floc explains the action of sand, gravel or coke trickling removal in conjunction with aeration treatments.

Generally speaking, aeration, contact with precipitated hydrate and organisms in a contact aerator, followed by filtration through sand, appears sufficient to remove manganese from most water. Soft waters, low in iron and pH value and high in new organic matter, present many difficulties. At Lowell, Mass., for instance, aeration to a high degree appears to form a resistant combination of manganese and organic matter. Hard waters, high in iron, appear relatively easy to demanganize. The present tendency is toward the upward flow type of contact aerator such as Pirnie(48) has in use at Long Beach. This type offers a longer period of contact in addition to saving in head and ease of cleaning. Storage before filtration, especially with gentle agitation, has been found to be helpful and even take the place of contact bodies. The plant at Brookline, Mass., where the trickler effluent passes through narrow channels at a moderate velocity to the filters, has given excellent results.

While manganese zeolite, regenerated with sodium or potassium permanganate, may be successful, chemical methods in common use are more frequently applied. As a rule, methods designed for iron removal, with allowances for slower oxidation of manganese and the maintenance of higher pH values in the treated water, will prove satisfactory in most cases. Zapffe(49) studied the fundamental principles involved and developed an experimental plant, where it was shown that manganese could be removed without bacteria or chemicals.

In a description of the process, he showed its application to the deposition of certain geological formations of large scale. It was believed that in the water mains bacteria begin depositing manganese which ultimately assumes the form of manganese dioxide. It is inferred that thenceforth manganese in solution is removed by inorganic catalysis under slightly alkaline conditions at pH values of 7.2 to 7.3.

The plant is based on this principle and provides for the use of pyrolusite (containing about 70 per cent manganese dioxide), coke, basic rock, and filtration. The water is forced upward through the finely ground pyrolusite and is thence sprayed on to a bed of small size coke resting on a thin layer of crushed basic rock. The coke beds remove 80 per cent of the iron and manganese by conversion of manganous hydroxide, formed by catalysis in the excess air or oxygen, to stable manganic hydroxide and finely divided manganese dioxide.

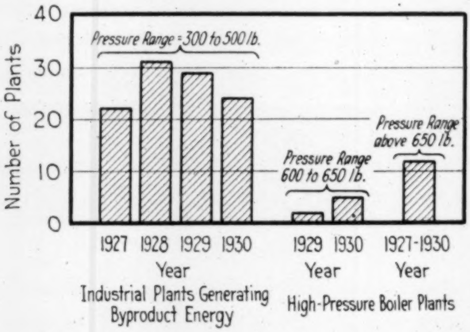

FIG. 6.—Showing Trend Toward Use of Higher Pressures and Temperatures in Steam Generating Units.

The open filter is built of crushed basic rock, to help promote alkalinity, and covered with fine silica sand. It is assumed that iron and manganese bacteria collect and propagate in the several units, providing for some biochemical assistance.

Difficulties have been experienced in filters of water treatment works through re-solution of removed manganese in idle filters and in the clogging of aqueducts and mains. Chlorination has practically solved these problems. This form of treatment is practiced at the Beverly Hills, Calif., water-softening and sulfur-removal plant for the elimination of "mud balls" produced by manganese deposition. The chlorine treatment disintegrated the mud balls, thus indicating that superchlorination destroys the growths associated with manganese deposition. Hale's(50) observations in 1928 reported chlorination as effectively removing and controlling manganese accumulation in New York City's aqueducts.

Recent experience in controlling chlorination of public water supplies has demonstrated the necessity of determining the manganese content of water in order effectively to control sterilization of such supplies and to afford the required protection against bacterial infection to domestic consumers. It is common practice to regulate chlorine dosage by maintaining specific amounts of residual or free chlorine in the treated water, then testing the chlorinated water with ortho-tolidine solution.

Manganese reacts also with the latter chemical, so that where manganese is present, modification in this essential test must be made to secure accurate results.

Manganese has been removed effectively from waters by means of manganese zeolites. These materials are primarily sodium zeolites which have been treated with manganous chloride to convert them into manganous zeolite. The reactions occurring may be written as follows as quoted from Appelbaum and Bretschger(51):

$$\begin{aligned} \text{“}\mathrm{Na_2O\cdot Z} + \mathrm{MnCl_2} &= \mathrm{MnO\cdot Z} + \mathrm{2NaCl} \\ \mathrm{MnO\cdot Z} + a\mathrm{NaMnO_4} &= \mathrm{Na_2O\cdot Z} + \mathrm{MnO\cdot Mn_2O_7} \end{aligned}$$

where Z indicates zeolite. The higher oxides of manganese formed in the second reaction are really carried by the zeolite on the surface of the grains. The zeolite proper acts as an active carrier and catalytic reagent. The oxides themselves do the work of iron and manganese removal. These oxides give up oxygen to the water for the necessary oxidation reactions, thus:

$$\mathrm{Na_2O\cdot Z} + \mathrm{MnO\cdot Mn_2O_7} + \mathrm{2Mn(HCO_3)_2} = \mathrm{Na_2O\cdot Z} + \mathrm{5MnO_2} + \mathrm{4CO_2} + \mathrm{2H_2O}$$

After the higher oxides have given up most of their available oxygen in this manner, they are reoxidized by passing permanganate solution through the bed. The amount of permanganate required is about 0.01 lb. per 1000 gal. for the average water."

Removal of manganese by the zeolite process has been fairly widely practiced both in this country and in Continental Europe but has not been used as extensively as other purification processes.

TREATMENT OF WATER FOR USE IN BOILERS

There has been a marked trend in recent years toward the use of higher pressures and temperatures in steam generating units. This is graphically illustrated in Fig. 6. Such conditions have focused attention sharply on specifications for satisfactorily processed water to cope with the critical condition imposed. This has necessitated drafting the services of the physicist and colloid chemist, the metallurgist, petrographer and of others in allied groups. Intensive operation of steam generating units has introduced many problems of scale formation, of little importance under less severe conditions. It is the purpose of feed water treating systems to eliminate scale formation on heating surfaces and to inhibit corrosion and embrittlement of the boiler shell. The result should be obtained without increasing the priming and foaming characteristics of the boiler-water salines.

The problem of deposition of boiler scales is primarily one of the solubility of salts at elevated temperatures. Although there are many data available on the solubility of salts in aqueous solutions at relatively moderate temperatures, there is a dearth of such equilibria data at elevated temperatures. Even when available it is seldom possible to relate the values of those occurring in concentrated boiler-water salines. The necessity for the collection of such data has been responsible for the inauguration of the several research programs now in progress for the solution of these and correlated problems.

The control of deposition of incrustations on heating surfaces of high-pressure boilers involves an intimate knowledge of thermodynamics and crystallography, and the influence of temperature, pressure and solubility coefficients of complex solutions on crystal formation. The most plausible explanation of the deposition of solids in boilers is that advanced by Dewrance(52) and later by Partridge(53):

The observations of the latter are quoted verbatim since they represent the most plausible explanation of this phenomenon: "Scale deposition and growth on the heating surfaces of boilers is due chiefly to the fact that certain slightly soluble substances become less soluble with increase in temperature. Some initial deposition of scale crystals by sedimentation, or by trapping in surface irregularities may take place in boilers of the horizontal return tubular or marine types, but it is probable that scale formation in the tubes of water-tube boilers is due to an entirely different action.

"The initial deposition of scale crystals is generally the result of crystallization at the boiler surface from a solution which is supersaturated with respect to the scale-forming substance. The whole body of a boiler solution constantly tends toward a state of supersaturation as steam is removed; in addition, since scale-forming substances decrease in solubility with increase in temperature, the film of boiler water adjacent to a heating surface tends to be supersaturated even when the body of the solution is only at the saturation point. Supersaturation in the liquid film at the heating surface may be relieved by spontaneous crystallization, possibly promoted by irregularities in the surface, even when no vaporization is taking place.

When bubbles are being evolved at the heating surface, however, the excess solute is deposited as minute crystals in rings directly at the solid-liquid-vapor interfaces formed by the metal surface, the boiler water, and the bubbles forming in contact with the surface. This latter action undoubtedly accounts for the deposition of practically all of the initial scale nuclei on boiler surfaces at which actual vaporization of water is taking place.

"The growth of scale following initial deposition of crystal nuclei may proceed in either of two ways. The existing crystals may grow by contact with the supersaturated liquid film with which they are continuously in contact. If these crystals are removed from contact with the solution, as by the adsorption of a film of a colloidal substance present in the boiler water, further deposition of new crystals superimposed on the previous deposit may take place as the result of bubble evolution. In the first case the resulting scale will consist chiefly of relatively large continuous crystals oriented more or less perpendicularly to the heating surface; in the second case, the scale will consist of relatively small crystals with a random orientation."

The growth of such deposits is undoubtedly accelerated by the effect of skin friction at the surface of the tubes, since the velocities along the tube wall are much less than toward the center of the tubes. This latter phase in the mechanism of scale growth has not been studied by investigators but the author's recent experiences would indicate that it has a marked accelerating effect and accounts for the deposition of many forms of silicate scale which might not otherwise develop in the presence of very small amounts of siliceous materials present in the feed water. Since scale laid down in boilers is the result of the deposition of minerals at high temperatures and pressures, the phenomena are comparable to geologic formation, confirmation of which is clearly apparent through petrographic identification. The application of petrographic procedure for critical studies of industrial water has revealed a close similarity between many sedimentary and igneous deposits occurring in nature and synthetic deposits in modern boilers.

Determining the composition of boiler scales by quantitative chemical analyses has always been, and still is, widely practiced. Such analytical procedure serves a useful purpose by determining the type and quantity of various constituents present in the deposits under examination but, beyond this, little accurate information is to be gained by such an analysis. It is impossible, from these data only, to determine the density or porosity of the deposits, the exact mineralogical classification of the material, or any other essential information relating to its formation, structure or physical properties. Such analyses are merely arbitrary mathematical combinations of the specific elements recorded. They may or may not indicate the type of material deposited, depending upon chance and the intelligence and experience of the observer. Such analytical reporting, unless confirmed by physical, microscopic or crystallographic examination, will give little specific information concerning crystal formation or variation of crystal growth of one type of deposit upon another. Chemical analyses of boiler scales are useful, primarily, for determining the presence of various elements or as supplemental to other physical or petrographic data. Beyond this, such analytical procedure is of little value.

Supplementing ordinary chemical analysis by petrographic studies will identify the crystallographic structure and reveal essential information about the development and physical properties of such deposits. By critical petrographic examination of crystal growths on boiler surfaces much specific information is obtainable as to the influence of the primary water-conditioning processes and the resulting solubility equilibria under the existing thermal conditions.

Petrography is the systematic and descriptive side of the study of rocks. This may be accomplished by microscopic examination of minerals of thin section or by determining the optical constant of the powdered specimen in a liquid medium, whose indices of refraction are known. Examination of scales in thin section requires careful preparation of the specimen. The sample to be examined is ground smooth on the surface and cemented to a microscopic slide in the Canada balsam. The exposed side is then ground and polished with successively finer abrasives until it is about 0.03 mm. thick and the faces are accurately parallel. The wafer is finally transferred to Canada balsam on a clean slide, and covered with a thin cover-glass. There are about twelve major specifications that characterize minerals (fewer in simple cases) and for the 1200 to 1500 known minerals these have been determined and tabulated. In practice the crystalline compounds encountered are less numerous, and the determination of a few properties may suffice. The petrographer applies these criteria in turn until the mineral is identified. The instrument used for identification is a specially designed high-grade compound microscope, with additional parts which make it possible to examine a sample in plane polarized light.

The immersion method consists of immersing the powdered mineral in a liquid medium whose indices of refraction are known and determining its optical constants.

The Characteristic Properties of Minerals:

The atoms and ions of crystalline solids are rigidly fixed in a sort of three-dimensional lattice, which has characteristic angles and dimensions. All crystals are made up of tiny geometrical forms which crystallographers have classified into systems according to their symmetry and to the number and disposition of their axes. The regularity of fine-structure is not often seen in large crystals because of environmental modification, but it gives rise to physical and especially optical properties that are perfectly definite. These include opacity, transparency and color, index of refraction, shape and form, and cleavage planes.

Observation of thin-section specimens, under low magnification, reveals a mosaic of geometrical shapes, needles, veins, and preserved fossils depending upon the mineral under examination. The identification of the structure and growth development of mineral deposits occurring at low or elevated temperatures is entirely practical by the petrographic method. Such procedure is obviously unduly costly and too time consuming for routine control. It has merit for the identification of deposits in high-pressure boilers, in turbine and superheater tubes or in other industrial conditions requiring special control. By correlation of such crystal growth with boiler water conditioning and operating data, procedures for inhibiting scale deposits may be developed.

There are few crystallographic data on complex boiler scales with the exception of studies of calcite, aragonite, anhydrite and brucite. Much is still to be learned concerning the nature and deposition of the many complex silicates and other minerals before accurate water-control procedure can be assured. To carry on such work intelligently requires trained workers and adequate microscopic equipment for identification and interpretation. Recent studies in this field have revealed many interesting and instructive crystalline forms which formerly defied accurate identification by other analytical procedure. Deposits removed from runners of a hot-water pump indicated on chemical analysis that they consisted primarily of calcium carbonate but without the characteristic appearance of aragonite or calcite. The material was very hard and porous, occurring in dark-brown laminated sections. By petrographic study in thin sections it was readily identified as calcite which was contaminated with opal, a hydrated silicate, and it contained also occluded organic matter. Serious tube difficulty developed from scales in a high-pressure boiler within one month after the installation was completed. The deposits, identified as laminated calcite, anhydrite and opal, caused much difficulty due to their porosity resulting in what amounted to steam blanketing with poor rates of heat transfer and localized overheating. Possibly the most interesting type of scale identified by petrographic analytical method is analcite. A description of this deposit, although reported elsewhere, is given here because of its unusual properties:

"This silicate is one of the zeolite groups having many interesting properties. According to Doelter(54) analcite, a double silicate of sodium and aluminum ($Na_2O \cdot Al_2O_3 \cdot SiO_2$) will form whenever the components exist in a closed system between temperatures of 180 C. and 430 C. (350 F. and 800 F.). The temperature of the boiler water from which this scale deposit was taken was 290 C. (550 F.), but the temperature of the tube surface was not known. Above the upper temperature limit of 430 C. (800 F.) analcite is said to pass into an hydrous compound (Nepheline) of the formula $Na_2O \cdot Al_2O_3 \cdot 2SiO_2$. Below the lower limit of 180 C. (350 F.) analcite passes into Natrolite, another zeolite, $2H_2O \cdot Na_2O \cdot Al_2O_3 \cdot 3SiO_2$.

"Analcite is not a rare mineral in regions of volcanic rocks. It is frequently found in fissures and cavities in lavas, often in fine crystals. Its origin is in the hot aqueous solution that marks the final phase of volcanic activities after the magmatic solutions have reached a moderate temperature and pressure. Artificially, analcite has been made in various ways, such as heating sodium hydroxide or silicate in a sealed aluminum glass tube at 180 to 190 C. (355 to 375 F.) for 18 hr.(55). Schulten(56) prepared synthetic analcite by mixing solutions of sodium silicate and sodium aluminate in such proportions that the alumina and silica were the same as in analcite, adding a suitable amount of lime water, and heating in a sealed copper tube at 180 C. (355 F.). After 18 hr. he obtained crystals of analcite of the following compositions:

	ARTIFICIAL ANALCITE	NATURAL ANALCITE
Silica, per cent	55.4	54.4
Alumina, per cent	22.1	23.3
Soda (difference), per cent	14.3	14.1
Water, per cent	8.2	8.2
	100.0	100.0"

It is to be noted that the lime water has no other function than to facilitate the crystallization. Viewed through a microscope the surface of analcite is seen to consist entirely of well-defined faces, showing typical cubes and trapezohedrons. The crystal faces comprising the upper surface of the scale are referable to the cube and the trapezohedron, the most frequent of the forms shown by analcite. At an early stage in the deposition of the scale, the cube is built up in skeletal crystals, these cubic edges are truncated by the trapezohedral faces, and these grow until the advanced stage shown in the last figure. This is apparently a state of crystallographic equilibrium, since there is no further advance to the simple trapezohedron by further replacement of cubic faces. These conditions are illustrated

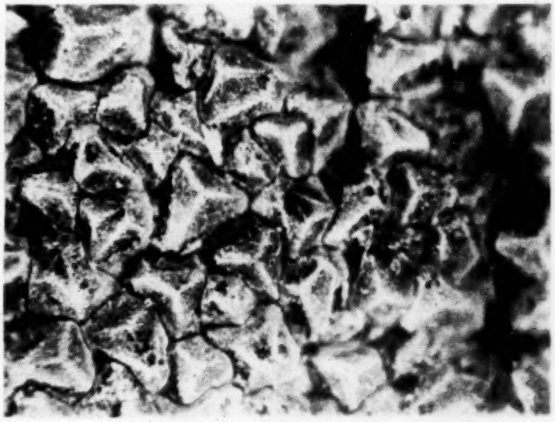

FIG. 7.—Photomicrograph of a Thin Section of Analcite Scale Showing Dominant Trapezohedral Faces (× 100).

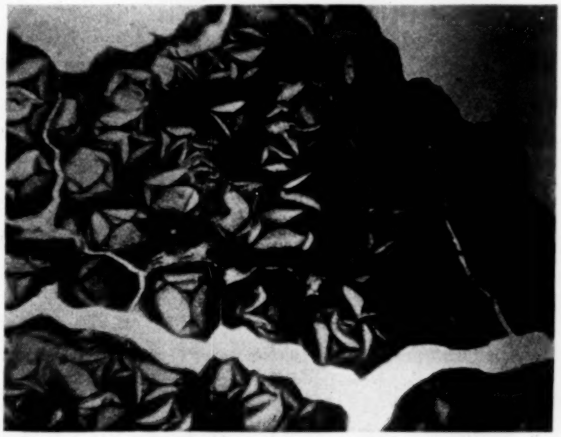

FIG. 8.—Photomicrograph of a Thick Section of Analcite Boiler Scale Parallel to Surface of Deposition of Scale, Showing Orientation of Submicroscopic Impurities at Intersection of Crystallographic Planes (× 100).

in Fig. 7. Charles Milton[1] in describing the condition of deposition states: "When the solution is very concentrated and alkaline, the crystals form trapezohedrons, under other conditions cubes." A typical example is shown in Fig. 8 showing dominant trapezohedral faces identified in thin sections. A different view of the same formation is illustrated in a photomicrograph of the same thin section parallel to the surface of deposition of the scale and shows orientation of submicroscopic impurities at the intersection of the crystallographic planes.

FIG. 9.—Dendritic Copper Crystals from Mud Drum Deposit (× 35).

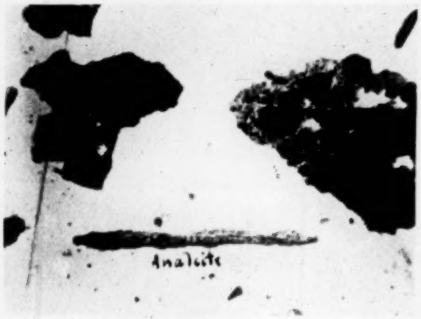

FIG. 10.—Analcite (Transverse Section) and Gypsum-Clay Aggregates from Superheater Tube Deposit (× 35).

Several growths of dendritic copper crystals were found in the deposit; the presence of which had previously been determined by chemical analysis amounting to 2.1 per cent of copper oxide. A reproduction of these copper crystals is shown in Fig. 9. It was believed that the crystals of metal copper were due to the reduction of copper salts, in the boiler-water salines, to a metallic state. The only apparent explanation for the presence of copper salts in the water was from the corrosion of brass fittings. It is conceivable that in contact with the iron surfaces of the boiler electrochemical deposition could occur with the deposition of metallic copper with an equivalent solution of iron.

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[1] Private communication.

At times these deposits are laid down as conglomerates, several deposits occurring simultaneously. These phenomena are clearly the result of solubility equilibria together with occluded substances. Such a deposit is shown in Fig. 10. This photograph was made of a thin section and shows gypsum-clay aggregates in the presence of typical analcite. The deposited scale was removed from a superheater tube.

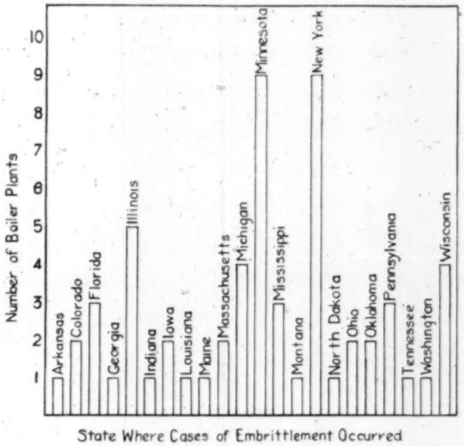

FIG. 11.—Showing Geographical Distribution of Boiler Plants Where Boiler Failed from Caustic Embrittlement Since 1930 as a Result of Improper Conditioning of Feed Water.

Influence of Concentrated Boiler-Water Salines on Cracking of Boiler Steel:

For more than twenty years the embrittlement of boiler steel by the use of unbalanced waters has been recognized and many failures from this action have occurred. Notwithstanding the wide distribution of data indicating the causes and correction of these difficulties, failures continue to occur. Since 1930 embrittlement of boilers has been detected in about sixty steam-generating stations involving a partial or total failure of approximately 175 boilers. The estimated cost of the replacement of or repairs to the equipment resulting from this form of cracking of steel has been not less than $800,000. A conservative estimate of the total value of the boiler equipment in the plants affected during the period noted is close to $5,000,000. Such losses have not been limited to any geographical section of the country, as is indicated by the distribution in the various states, shown in Fig. 11, nor have they been restricted to any type of water conditioning, but have occurred from all types of waters where the concentrations of sulfates in the boiler-water salines has been too low to furnish the necessary protection against caustic attack.

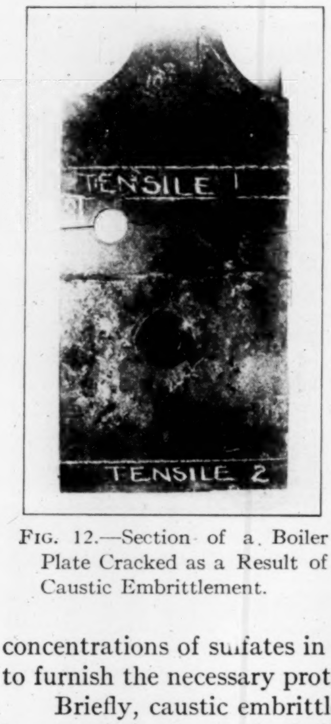

FIG. 12.—Section of a Boiler Plate Cracked as a Result of Caustic Embrittlement.

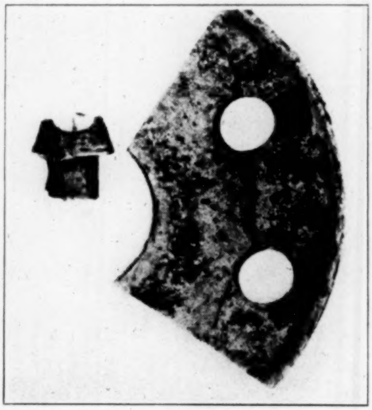

FIG. 13.—Section of Mud Drum Blow-Down Flange, Cracked as a Result of Caustic Embrittlement.

Specimen cleaned in alkaline solution of ammonium citrate.

Briefly, caustic embrittlement is the destructive chemical attack by caustic soda on the boiler steel, under stressed conditions, and may occur when boiler-water salines contain high concentrations of caustic soda and relatively low concentrations of sulfates. The failure of steel occurs in areas of greatest stress. All cracks that occur in boilers are not due to the use of poorly conditioned water, but where cracks develop the cause should be definitely allocated and the conditions thoroughly investigated. The prevention of this form of destructive action is so relatively simple that it is difficult to conceive why these costly failures should continue to develop. Adequate protection will be effected by the maintenance at all times of specific ratios of sodium sulfate to total alkalinity. The desired ratios vary with the boiler pressures maintained, ranging from one of total alkalinity to one of sodium sulfate up to 150-lb. pressure, to as high as three of sodium sulfate to one of total alkalinity in the boiler-water salines above 250-lb. pressure. Although experience and extensive research has definitely indicated that water conditioned as indicated will provide the necessary protection, the mechanism of the protective action is still somewhat vague. The experimental studies begun in March, 1933, and still in progress at the Non-Metallic Station of the U. S. Bureau of Mines, at New Brunswick, N. J., were undertaken for the purpose of determining specifically the function of sodium sulfate in preventing corrosive attack of the steel. This investigation, and similar studies conducted elsewhere since 1912(57), are primarily for the purpose of establishing the solubility equilibria of sodium sulfate in the presence of caustic soda and in other concentrated boiler-water salines. To date the complete solubility curve of sodium sulfate has been determined. This research has indicated that sodium sulfate may exist in four forms between 0 and 350 C. (32 and 660 F.). The regions of stability and their solubilities has been established.

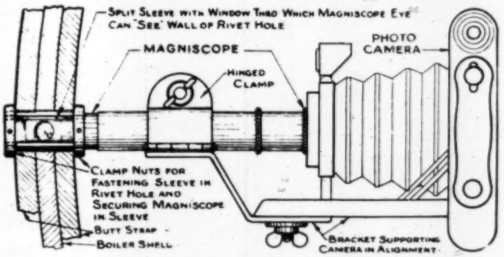
Split Sleeve with Window Thro Which Magniscope Eye Can "See" Wall of Rivet Hole
Magniscope
Photo Camera
Hinged Clamp
Clamp Nuts for Fastening Sleeve in Rivet Hole and Securing Magniscope in Sleeve
Butt Strap
Boiler Shell
Bracket Supporting Camera in Alignment

FIG. 14.—The Hartford Magniscope Rigidly Mounted in a Rivet Hole by Means of a Threaded Sleeve and Clamp Nuts.

The sleeve has an aperture through which the eye of the magniscope sees the surface of the hole. The camera rests on a bracket which affords accurate alignment.

The destruction of steel by embrittlement is insidious and frequently when discovered has advanced so far as to require abandonment of the boilers. If the caustic action is limited to riveted holes and discovered in time, the holes may be reamed and over-size rivets used. Frequently, however, steel has been so seriously damaged as to require complete replacement of the boiler drums or the entire unit. The characteristic appearance of steel subject to caustic embrittlement shows fine hair cracks radiating into the metal from the periphery of rivet holes (Figs. 12 and 13). In the advanced stages the cracks may carry continuously from one rivet hole to another. In one installation, examined recently, a section of plate was cracked for a distance between 48 rivet holes.

It is a costly undertaking to dismantle completely a boiler suspected of damage by caustic embrittlement. It is customary where embrittlement is indicated to remove one or more rivets and to examine carefully the suspected damaged area of the plate. An ingenious appliance[1] for making such an inspection has been developed and put into practical use. The apparatus, shown diagrammatically in Fig. 14, consists of a magnifying periscope attached to a suitable camera. For examination, the section of plate at the rivet holes is thoroughly cleaned and etched with acid. The section under examination is illuminated by a flashlight and if cracks are noted they may be

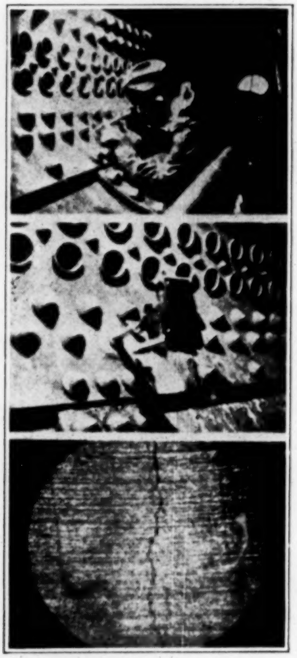

FIG. 15.—Examination of Boiler Steel by Means of the Magniscope.

Top—Locating a crack.
Center—Photographing.
Bottom—The Photograph.

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[1] Designed by J. P. Morrison, Assistant Chief Engineer, Hartford Steam Boiler Inspection and Insurance Co., Hartford, Conn.

photographed. Reproduction of a cracked plate and the position of the camera in place for operation is shown in Fig. 15.

Corrosion of Iron and Steel in Steam Generating Stations:

The control of corrosion of ferrous and non-ferrous metals in all steam stations is a major problem requiring intimate knowledge of the fundamental causes of such action. So many factors influence corrosion that there can be no single remedy, and each case must be diagnosed and controlled with regard to local conditions. Corrective measures depend, therefore, on (*a*) an intelligent analysis and evaluation of the factors involved, and (*b*) specific correction and control of conditions responsible for the losses sustained.

Many theories have been advanced to account for the corrosion of metals. The electrochemical theory, now generally accepted, was suggested about thirty years ago by Whitney(58) and has since been confirmed by experience and subsequent investigation. Briefly stated, the explanation is, that(59):

"All metals in contact with water have a specific solution tension, that is, they exert the power to go into solution. When this reaction takes place, the metal that has dissolved in the liquid is in the form of ions.[1] This action may take place when the metal and water are chemically pure. Under these conditions, the rate at which the metal dissolves is very slow. The solution tension is influenced greatly by impurities in the metal and in the electrolyte. The solution of pure iron in water, according to this theory, takes place in the following way:

"Iron dissolves only by displacing another metal which is already in solution in the electrolyte. This may be noted by submerging iron in a copper solution. When this is done, the iron goes into solution in the electrolyte and the copper is deposited upon the iron.

"Water (H_2O)[2] when ionized forms hydrogen ions H^+ and hydroxyl ions OH^-. The iron in contact with the water goes into solution as ferrous ions, and an equivalent number of hydrogen ions (the hydrogen being considered a metal) are discharged and plated out upon the surface of the iron. The ferrous ions in contact with the OH ions form ferrous hydroxide. In the presence of oxygen, ferrous hydroxide is oxidized to ferric hydrate.

"When the hydrogen deposits upon the surface of the iron the metal is polarized, and no further corrosion of the iron results until the layer of this gas is removed. In the presence of oxygen, the hydrogen is removed by combining chemically with the oxygen to form water. The hydrogen may be removed also by passing away as gaseous hydrogen. In these ways, the hydrogen is removed as it forms and the corrosion is progressive.

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[1] Atoms or atom groups carrying charges of static electricity. If the ions have a positive charge, they are termed "cations," and if they carry a negative charge, "anions."

[2] For the purpose of illustration, the formula for water may be written HOH which ionizes to H^+OH^-.

"In the presence of oxygen, the iron that has gone into solution in the water in contact with the metal is readily oxidized, forming insoluble compounds. As these deposits occur, the corrosive action is somewhat retarded, since they act as a protective coating to the metal. Corrosion of iron is not entirely prevented, however, and corrosion may and usually does proceed beneath rust nodules (60).

"Practically all natural waters contain oxygen and as such are corrosive to a greater or lesser degree. The rate of solution is affected by the impurities in the metal and in the electrolyte. Because of these facts, the solution tension is considerably different at different portions of the material. The solution tension is increased in some areas and decreased at others. Due to these conditions, electric currents may flow from one area to another, the electrolyte serving as the conducting medium. This is equivalent to the action of an ordinary wet-cell battery. The metal passes into solution in the electrolyte at the electropositive area (the anode), the hydrogen ions migrating to the negative areas and the hydroxyl ions to the positive area."

From the foregoing discussion it is obvious that dissolved oxygen exerts a marked effect in accelerating corrosion rates.

High concentrations of hydrogen ions tend to increase the corrosion of the metal due to the fact that an increase of hydrogen ions raises the acidity of the solution. This in turn accelerates the deposition of atomic hydrogen, and since atomic hydrogen deposits as iron ions go into solution, it follows that for each ion of metal which passes into solution, there is deposited or plated out one electric equivalent of hydrogen. Any agent which increases the rate of deposits of hydrogen on the metal permits an increase in the solution of the metal. This phenomenon is influenced by a great variety of factors, the importance of which varies under local conditions. Summarizing the results of experience and existing knowledge of corrosion phenomenon, the two most important factors for the inhibition of active attack of boilers is the control of the pH value of the feed water and concentrated boiler-water salines, and the completeness of deaeration of the feed-water supply. The establishment of maximum pH values and minimum dissolved oxygen concentrations of water which will insure complete inhibition of corrosion is difficult, if not impossible, since many other factors influence this phenomenon. If, however, the residual dissolved oxygen in feed water is limited to a maximum of 0.01 ml. per liter and the pH value of the water is maintained at 9.6 or higher, active corrosion will be retarded. Unfortunately, such control may not always be effected owing to the limitations of the available equipment and the specific operating conditions encountered.

Reduction of Corrosion Losses by Deaeration of Water:

There has been a marked trend in recent years toward better oxygen removal from feed water by the development of deaerating heaters. Such equipment is now considered essential in all modern steam-station practice. Unfortunately, our analytical methods for determining small amounts of oxygen in water have not kept pace with other developments in the art. It is frequently difficult to diagnose accurately the primary causes of corrosion and the influence of dissolved oxygen in small amounts on account of faulty analytical procedures. It is doubtful whether oxygen in amounts less than 0.01 ml. per liter can be determined accurately by the existing analytical test procedures, so that it is difficult to determine the completeness of deaeration effected even in the best type of deaerating heater.

Supplementary deaeration to remove the last traces of oxygen by chemical compounds is now attracting attention. The deaerating chemicals most used are ferrous salts, tannin compounds and sulfites. These chemicals have merit for clearing up the last traces of oxygen, but our knowledge of the chemistry involved, especially in reference to the velocities of the reaction, is limited. Recent investigation has demonstrated that the removal of the oxygen by sulfites is not directly proportional to the amounts indicated by the theoretical chemical reaction. It has been demonstrated that two or three times the amount of sulfite over the theoretical combining reaction may be required to remove a given amount of oxygen. Further, the reactions, contrary to general assumption, are not instantaneous. The problem of determining the efficacy of oxygen removal by sulfites is complicated, since at the present time there are no simple and accurate methods of analysis for determining dissolved oxygen in the presence of this chemical.[1]

A few steam stations using supplementary sulfite treatment for complete deaeration have reported satisfactory results. Removal of oxygen by sulfite or other chemicals is not advised as a substitute for well-designed open or deaerating heaters. Chemical deaeration should be considered merely as a supplementary form of treatment for the final removal of traces of oxygen and should only be applied where mechanical deaeration is not sufficiently complete to avoid accelerated corrosion.

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[1] Two methods for the determination of dissolved oxygen in the presence of sulfite will be found in *Zeitschrift für analytische Chemie*, Band 90, Heft 7 and 8 (1932). These articles are entitled "The Determination of Oxygen in the Presence of Sulfite" p. 241, by L. W. Haase, and "The Determination of Residual Oxygen, in Deaerated Sulfite Containing Feed Water, with the Help of a New Simple Apparatus," p. 246, by W. Wesley.

Unaccountable corrosion losses may occur even after efficient deaerating equipment is installed. Frequently, these conditions are the result of reentrainment of oxygen beyond the deaerator.

At times erratic oxygen results in feed water will be obtained although the deaerating equipment is functioning at a maximum efficiency. The trouble may be traced to the intermittent discharge of bubbles of air which accumulate slowly at the high point on the pump casing. In order to assure accurate sampling, it is necessary to remove trapped air by opening the pet cocks at the top of the casing above the individual stages of the pump.

Although the determination of dissolved oxygen by the Winkler method is relatively simple, accurate analysis requires particular care in the technique employed, especially where it is necessary to determine oxygen in amounts less than 0.03 ml. per liter. The following precautions should be taken in order to insure accurate results:

1. Only chemicals free from oxidizing impurities should be used.
2. Water for making test solutions should be free from nitrites, sulfites, organic matter, ferrous or ferric iron.
3. Samples should be drawn through copper tubing and all joints should be brazed wherever possible.
4. Sampling cocks and unions should be made tight by shellacking.
5. New lines should be flushed out 24 hr. before using.
6. Red lead used for making joints tight will introduce a large error. Samples which must be drawn through such a pipe line should be flushed out for one or more days before the test is made.
7. Water containing 1 p.p.m. or more of ferrous iron or 0.1 p.p.m. or more of nitrite nitrogen should be tested by the Rideal-Stewart modification(61) method.
8. New rubber tubing should not be used, since appreciable errors are introduced from dissolved sulfur.

To what extent corrosion losses in high-pressure boilers occur as a result of direct oxidation by decomposition of steam is difficult to determine without special study. Such losses probably do occur where high superheat is developed. It is well known that black oxide of iron is formed when steam is dissociated. This takes place according to the following reaction:

$$\begin{array}{ccccccc} 4H_2O & + & 3\,Fe & = & 4\,H_2 & + & Fe_3O_4 \\ \text{(Water)} & & \text{(Iron)} & & \text{(Hydrogen)} & & \text{(Magnetic Oxide of Iron)} \\ \text{(Steam)} & & & & & & \end{array}$$

There have been only few studies of this kind but observers indicated that such action is likely, although not of serious consideration. Below 750 F. (400 C.) the volume of gas production by dissociation of water is probably negligible. At higher temperatures, conditions may occur in which hydrogen gas is evolved at a relatively rapid rate indicating active corrosion. Fellows, reporting the results of experiments on "The Dissociation of Water in Steel Tubes at High Temperatures and Pressures,"(62) drew the following conclusions:

1. The rate of decomposition of metal increases as the temperature of the surface over which the steam passes is raised.
2. The rate of decomposition of metal decreases as the thickness of the oxide formed on the surface increases. There is no evidence that this oxide attains sufficient thickness or imperviousness to completely protect the metal from further oxidation.
3. For practical consideration the variation in steam pressure within the limits used appears not to affect the rate of decomposition of the metal over which it passes.
4. The decomposition of low-carbon steel under the conditions of the test proceeds at a much faster rate than that of chromium-steel used under the same conditions.

Fellows further states:

"Expressed very roughly we find at steam velocities approximately 3000 ft. per min., no hydrogen, no dissociation and consequently no oxidation of the metal from this cause. At velocities approximating 850 ft. per min. small quantities of hydrogen become evident and finally at velocities of the order of 25 ft. per min. we get relatively large quantities of hydrogen and severe corrosion of the low-carbon steel as used in these tests and of which superheaters are made. Since velocity of steam is an important factor in governing the metal temperature such observations indicate the probability of a very definite relation between velocity of steam and oxidation of metal through dissociation of steam." These observations would indicate the desirability of similar studies in high-pressure steam stations encountering corrosion losses not readily attributable to poor feed-water conditioning or other observed corrosion factors.

It is fairly well known that dissimilar metals in contact greatly accelerate rates of corrosion resulting in localized attack of the baser metal. For instance, when copper and steel are in contact the steel will be aggressively corroded with practically no loss of copper. The rate of the attack will depend on the hydrogen-ion concentration of the electrolyte (the solution in contact with the metals), the dissolved oxygen present, and other factors. Since the composition of mill scale is different from that of steel and iron, mill scale in contact with ferrous metal frequently sets up localized corrosion. Mill scale probably is responsible for greater localized corrosion than is experienced generally from other dissimilar metals in contact. Mill scale, which consists largely of magnetic iron oxides, is not readily attacked, and when it covers the entire surface of steel serves as an excellent protective coating.

The difficulty arising from mill scale is from patches of scale in contact with areas of steel not so protected. Actually what happens is that the two materials are in electrical contact, and in the presence of an electrolyte an electric circuit is completed. When this occurs the nobler metal, namely, the mill scale, becomes cathodic while the steel becomes anodic. When such a condition is set up, the mill scale or cathodic pole is protected, since it is plated with atomic hydrogen, while the other metal, in this case the steel, passes into solution. When such action takes place in boilers or appurtenant equipment, the iron or steel is aggressively attacked in the form of pitting, the corrosion taking place in exposed spots between the mill scale.

To avoid this form of corrosion, mill scale should be removed prior to putting the equipment into service. Removal of mill scale can be effected by pickling with acid or by mechanical means such as sand blasting. In recent years the process of removing mill scale by acid has been improved by the use of inhibitors.

In view of the marked influence of mill scale as a factor in corrosion, it is surprising that more attention is not given to the preparation of the surfaces of metal in boilers and appurtenant equipment where the metal is in contact with electrolytes (water) containing salts in solution having high electric conductance. More attention to this particular phase of corrosion activity would greatly lessen corrosion problems in steam generating stations.

SPECTROGRAPHIC ANALYSIS OF WATER

Accurate quantitative estimation of elements by spectrographic methods is the most delicate analytical procedure yet developed. Briefly, spectrographic analysis is the identification of substances by the light that they radiate or absorb, and an estimation of their quantity from the intensity of these phenomena. Expressed somewhat differently, quantitative estimation of elements is possible since up to a certain amount, which varies with different elements, "the intensities of the characteristic lines of an element are proportional to the relative amount of it present in the substance; and that these lines will practically disappear, becoming fainter and fainter, as the amount of the element in the light source is reduced."

To record these phenomena the spectrograph is used. This is an optical instrument which breaks up the light from a source into its constituent wave-lengths, and spreads the colors across a photographic plate in a form suitable for study. To produce the emission of radiation, substances must be volatilized by heat, decomposed into atoms, and the atoms distorted or partially decomposed, either by heat or electricity. The significant fact for the analyst is that the spectrum of radiation thus obtained from any element or compound contains a perfectly definite series of lines which are characteristic of that element or compound.

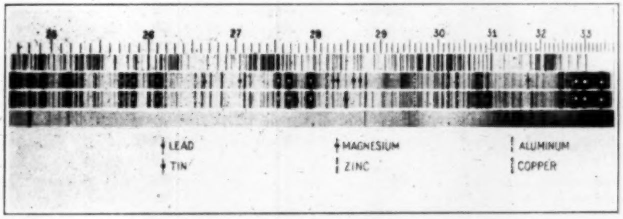

FIG. 16.—Spectrum of Alloy.

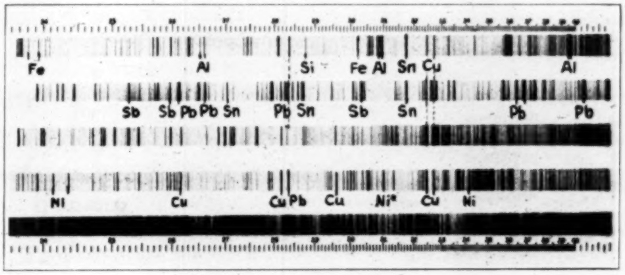

FIG. 17.—Spectrum of Alloy.

The intensity of spectral lines, under constant conditions of exciting energy, depends on the concentration of the atoms which generate the radiation. A bunsen flame will excite the spectra of a few of the softer metallic elements, but for many metals the electric arc is required. The emission spectrum method is adapted to qualitative or quantitative determination of elements and a few compounds in amounts as small as a few millionths of a milligram.

The principle involved in the quantitative estimation of an unknown material by the emission method is that under constant exposure conditions, the number and intensity of the lines of an element are proportional to its concentration in the sample (Figs. 16 and 17). This is valid only for small limiting concentrations which are different for each element. The spectroscopist compares th

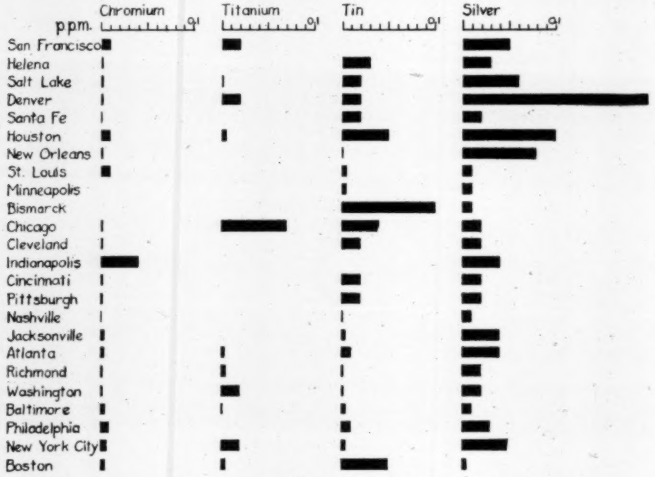

FIG. 18.—Rare Elements Identified in Public Water Supply by Spectrographic Analysis.

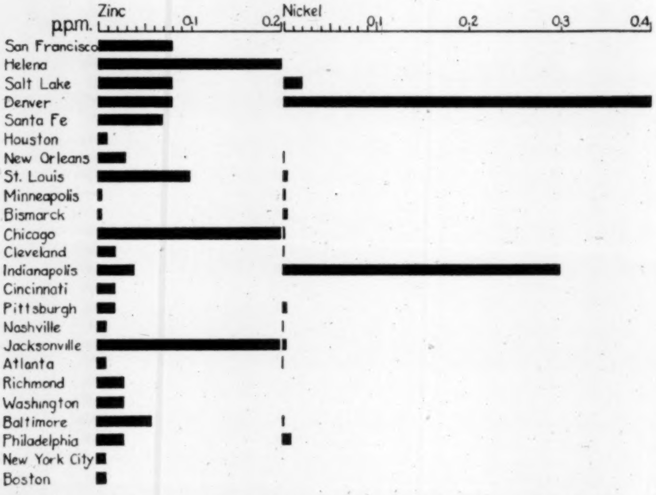

FIG. 19.—Rare Elements Identified in Public Water Supply by Spectrographic Analysis.

intensity of lines on the plate and translates them into concentrations. Such testing is, of course, beyond the scope of routine analytical work at present, but has the greatest potential value where accurate and complete analytical information is required. Its value for critical studies of municipal or industrial water supplies is evident.

Until the recently published studies of Braidech and Emery(63) appeared, few chemists or engineers, to say nothing of the general public, realized that many of our municipal water supplies contain lead, fluorides, and zinc in appreciable amounts, and that nickel, silver, boron and other metals were also detectable in a number of drinking water supplies, when the methods used for identification of these elements were sufficiently delicate.

The Braidech and Emery spectrographic study of public water supplies has revealed many interesting facts in reference to their chemical composition (Figs. 18, 19 and 20). For instance, silver was identified in traces in all water samples examined and occurred in appreciable amounts in the waters supplied to New York City, Atlanta, New Orleans and Denver. The water supplied to New York and Atlanta contained more silver than copper. The samples from Lake Michigan contained between 0.2 and 0.3 p.p.m. of zinc, incidentally, the highest quantity noted in any of the samples tested. Chromium was noted in all waters studied and likewise nickel was identified as spectrographic traces in all supplies excepting those of Denver and Indianapolis. In water supplied to both of the latter cities it was noted in amounts of from 0.3 to 0.5 p.p.m. It is of interest to note that nickel in Denver water exceeded the quantity of iron present. The quantities of manganese in many of these supplies is of considerable

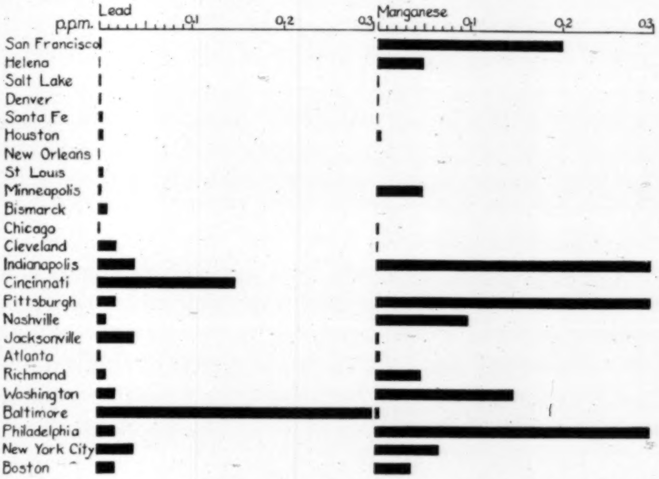

FIG. 20.—Rare Elements Identified in Public Water Supply by Spectrographic Analysis.

economic importance in respect to many industrial uses where manganese is to be avoided. From a sanitary viewpoint, the presence of lead in such relatively large amounts in many city water supplies, especially Baltimore, demands more than passing interest.

It is interesting to note that, in at least one instance, traces of lead in a number of public water supplies were shown definitely to have resulted in endemic lead poisoning among consumers of such waters. The findings reported were the result of an investigation conducted by the Metropolitan Life Insurance Co., and are sufficiently pertinent to warrant reciting here:

"Of 90 water sources used by the persons studied, 35 caused poisoning as determined by certain criteria. Of 253 exposed persons, 63 (24.9 per cent) were poisoned."

Further poisoning occurred among 14 persons ingesting as little as 0.1 mg. of lead daily over an average period of 8¼ years. These conclusions are highly important when considering the appreciable quantities of lead in the water supplies of Baltimore, Cincinnati, and other large cities. It is improbable that any of these substances have an appreciable effect in the majority of industrial processes and, from a purely engineering viewpoint, may be only of academic interest. It illustrates, however, that many compounds in water, even though they are present in infinitely small quantities, may have a far-reaching effect in special cases.

The adaptability and usefulness of spectrographic analyses of industrial water supplies will be limited to special problems when the presence of minor quantities of elements affect processes detrimentally. In this group are the dyeing and textile industries, manufacturers of high-grade paper, and certain refined chemicals and pharmaceuticals. Such data would add greatly to our knowledge of the specific chemical characteristics of high-pressure steam samples and condensate which up to the present is incomplete.

Water softening by chemicals and by base-exchange minerals has not been considered in the present discussion. Although these processes for conditioning industrial water supplies are of importance, they present no unusual operating difficulties, since the basic principles of control are well understood and water softening, generally, presents no unusual problem.

In this lecture it has been possible to do no more than to indicate certain trends in analytical procedure, since such procedure applies to critical studies of water as an engineering material. If our remarks have stimulated new viewpoints as to the importance of these problems, we have accomplished our purpose.

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TESTING SHEETS FOR BLUE BRITTLENESS AND STABILITY AGAINST CHANGES DUE TO AGING

By R. L. Kenyon[1] and R. S. Burns[1]

Synopsis

The authors have applied the principle that strain aging and blue brittleness of ferrous materials are but different manifestations of the same phenomenon and have developed a relatively rapid method of evaluating the aging properties of such material by determining the tensile strength at room temperature and at a temperature in the blue-brittle range. The apparatus used in the determination of the tensile properties of sheet material at elevated temperature (up to 600 F.) is described. The difference between the tensile strength at room temperature and that determined at 400 F. is correlated with the change in tensile properties of cold-rolled sheets due to an artificial aging treatment. The results indicate a quantitative relationship between these two properties.

Introduction

Many different tests have been used to follow the effects of aging and blue brittleness. They all have their advantages and disadvantages, but some of them are far superior in the ease with which they can be carried out and in the significance of the results obtained. These various considerations cannot be discussed here in detail, but after weighing them all carefully, the authors believe that the static tension test offers the most significant information. This is particularly true in connection with the inspection of ferrous sheet materials for deep-drawing purposes, because it is the only test that gives a quantitative measure of the tendency to stretcher strain.[2]

The static tension test has been widely used to follow the blue brittleness of ferrous materials, and it is also capable of following the changes in properties due to aging. It has long been known that the tensile strength of mild steel is greater at slightly elevated temperature than at room temperature.[3] Fettweiss[4] was perhaps the first to point out the connection between this behavior and the changes in properties due to aging.

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[1] Supervising Research Engineer, and Research Engineer, respectively, The American Rolling Mill Co., Middletown, Ohio.

[2] R. L. Kenyon and R. S. Burns, "Autographic Stress-Strain Curves of Deep Drawing Sheets," *Transactions*, Am. Soc. Steel Treating, July, 1933.

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[4] F. Fettweiss, "Blue Brittleness and Aging of Iron," *Stahl und Eisen*, January 2 and 9, 1919.

The rate at which the changes due to aging take place depends largely upon the aging temperature, other things being constant. Figure 1 shows the chronological change in tensile strength of mild steel cold-stretched 5 per cent and aged at various temperatures, as determined by Köckritz.[1] The higher the temperature, the more rapid the change. This has led most investigators to believe that the increase in tensile strength at an elevated temperature is due to instantaneous aging, the deformation in the test itself furnishing the necessary "cold work." The maximum tensile strength of mild steel occurs at about 570 F. (300 C.), although its exact location depends on the speed of testing.[2] Since this work was done, many attempts have been made to discover the mechanism of this transformation, but

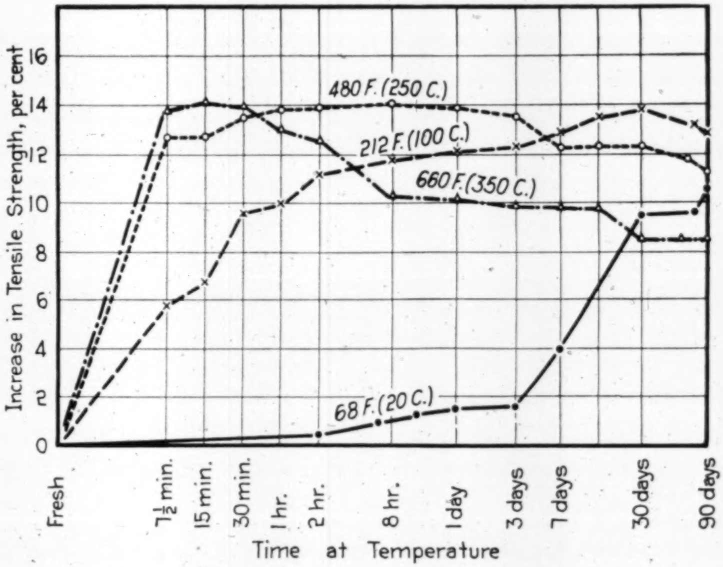

FIG. 1.—Chronological Change in Tensile Strength of Mild Steel Cold Stretched 5 per cent and Aged at Various Temperatures (Köckritz).

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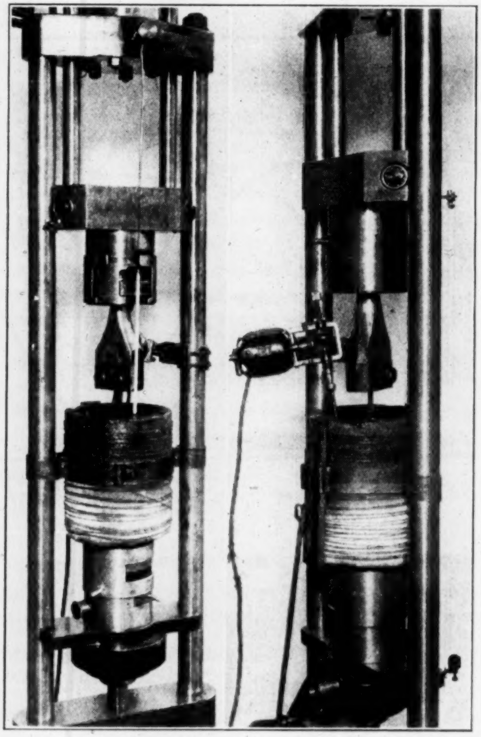
[1] H. Köckritz, "Über den zeitlichen Verlauf der Alterung weichen Stahles und über die Alterung von Stählen verschiedener Herkunft," *Mitteilungen* aus dem Forschungs-Institut der Vereinigte Stahlwerke, A. G., Vol. 2, No. 9 (1932).

[2] Le Chatelier explained this by assuming that cold working of iron or steel starts an irreversible transformation which requires a certain amount of time for its completion. The higher the temperature, the more quickly the transformation is completed. From 570 F. (300 C.) upward, however, there is a reduction due to the annealing effect, which also requires a certain time, decreasing with rising temperature, for it to become fully effective. Fettweiss summarized this by the following statement: "At temperatures under 570 F. (300 C.) the steel is found to be weaker and tougher the more rapidly it is tested, and above 570 F. (300 C.) the more rapidly the test is carried out, the more the annealing effect is masked and the steel appears to be stronger and more brittle." (From F. Fettweiss, *loc. cit.*)

there is still a question as to its exact nature. Nevertheless, there is now no doubt about the relationship between aging and blue brittleness. It is, therefore, entirely feasible to use a test for blue brittleness as a measure of the stability of the material against changes due to aging. The principal advantage of this lies in the elimination of a number of variables that have to be controlled in the case of aging tests. In aging tests, it is necessary to cold-work the material a definite amount, and then hold for a definite length of time at a certain temperature. Each of these variables—amount of cold work, time and temperature of aging—greatly influences the final results. Some materials change faster than others, therefore, the arbitrary selection of a particular time and temperature makes it difficult to compare the results obtained on different materials. In the case of the tests for blue brittleness, the difference in the physical properties



(*a*) Front View. (*b*) Back View.

FIG. 2.—Apparatus for Making Tension Tests at Elevated Temperatures on Sheet Specimens.

at room temperature and in the blue-brittle range furnish the criterion. It is only necessary then to establish a quantitative relationship between the two tests.

METHOD OF TESTING

With thin sheets there are many difficulties encountered in making, at elevated temperatures, even such a well-standardized test as the static tension test. The chief difficulties include: The method of heating the specimen uniformly in a manner to prevent excessive oxidation of the thin strip, measurement of the actual temperature of the specimen, gripping the specimen, and providing an autographic extensometer so that stress-strain curves can be drawn. Thus far we have been successful in making tension tests on thin strips (as thin as 0.015 in.) at temperatures up to about 600 F. (315 C.). The upper temperature limit depends largely on the flash point of the oil used in the heating pot to be described later.

Two views of the apparatus, which was designed for use in an Amsler testing machine, are shown in Fig. 2. The sample is held in special grips, the upper one of which can be seen in Fig. 2. The two $\frac{3}{4}$-in. cap screws on each grip are drawn very tight to keep the samples from slipping, and with a little practice it is possible to draw them down evenly so as to get uniform gripping across the specimen. The faces of the grips are serrated transversely with very fine shaper tool marks. The lower grip has a T-slot on its under side, which slips over a stud that is machined on the end of a long threaded shank that extends through the bottom of the pot and is fastened by means of a spherical seat and split ring to the lower or stationary head of the testing machine. The shank is fastened to the pot by means of two nuts that are properly packed and drawn tight to prevent oil leakage. The upper clamp can be disconnected from the movable head of the testing machine by releasing the split rings; this clamp also operates through a spherical seat.

An oil bath is used for heating the specimens, because it lends itself to closer temperature control than a vertical tube furnace and the specimens are brought to temperature more quickly. The construction of the pot is quite simple. It consists of a brass shell insulated with mica and wound with a nichrome heating element which is covered on the outside with asbestos insulation 1 in. thick. An iron-constantan thermocouple is silver-soldered to the inside of the brass shell about halfway up the side, and this is connected to a Leeds & Northrup potentiometer recorder-controller for regulating the pot temperature. The small stirring motor keeps the oil rapidly circulating and so minimizes the temperature gradient from the outer shell to the specimen in the center. As a matter of fact, by exploring the bath with a mercury-glass thermometer, the oil around the specimen was found to be within ± 1 F. (± 0.6 C.) of the temperature of the brass shell. A dummy specimen was prepared with a thermocouple silver-soldered to the midpoint of its gage length, and it was found that it came up to the bath temperature in less than one minute.

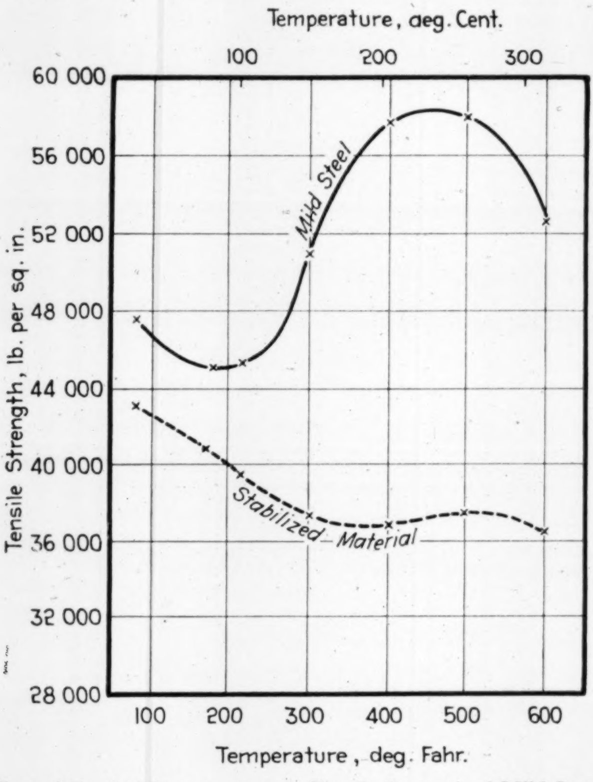


FIG. 3.—Effect of Testing Temperature on Tensile Strength of Mild Steel and New Stabilized Steel Sheets.

In order to obtain satisfactory results in any method of testing, it is necessary that the proper technique be followed; this is especially true in the case of tension tests at elevated temperatures. In making these tests, it is quite necessary always to use the same testing speed because the tensile properties are much more influenced by this variable at elevated temperature than at room temperature. All of the tests reported here were made with a machine cross-head speed equivalent to a rate of strain of 0.05 in. per in. per min. below the yield point, and 0.20 in. per inch of gage length per minute above the yield point. It is also true that the testing temperature must be very carefully controlled and kept uniform over the entire specimen.

RESULTS OF TENSION TESTS ON SHEETS AT ELEVATED TEMPERATURE

The tensile strength of ordinary mild steel sheets is influenced by temperature in a manner similar to that reported by various investigators[1] for other types of specimens of such materials. The solid line in Fig. 3 shows the results of tests on No. 19-gage mild steel deep-drawing sheets for automobile bodies. The strength is slightly less at 170 and 212 F. (75 and 100 C.) than at room temperature, but at 300, 400, 500 and 600 F. (150, 205, 260 and 315 C.) it is above that at room temperature. There is a maximum between 400 and 500 F. (205 and 260 C.). This differs from the maximum reported by LeChatelier and Fettweiss (300 C., 570 F.), but, as mentioned by the latter, this is caused by difference in testing speed.

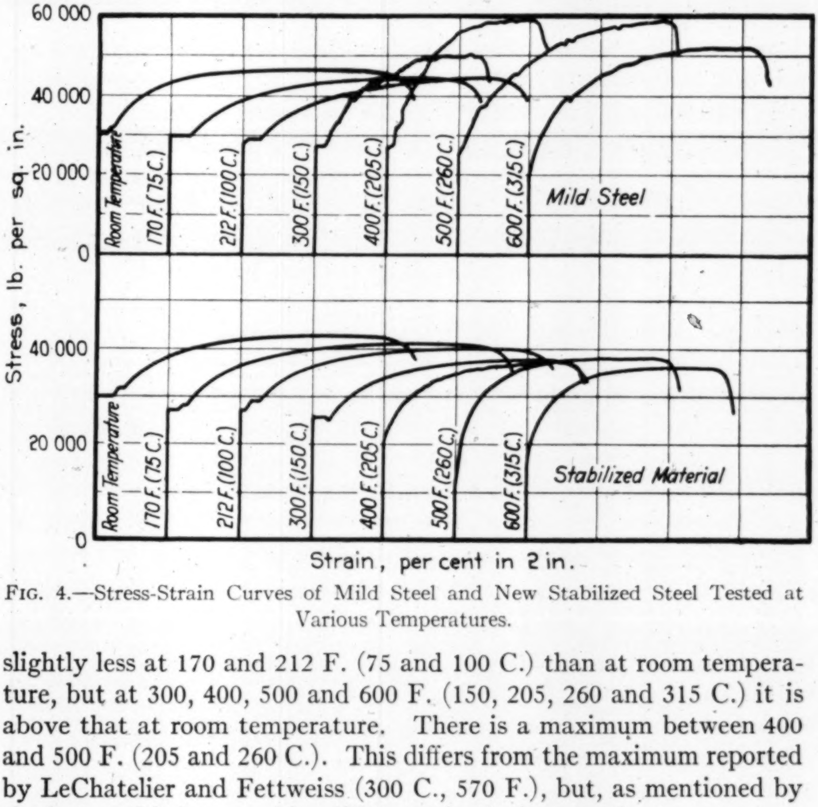


FIG. 4.—Stress-Strain Curves of Mild Steel and New Stabilized Steel Tested at Various Temperatures.

The stress-strain curves for this material, made at various temperatures, are shown in the upper part of Fig. 4. Those made at

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[1] F. Fettweiss, "Blue Brittleness and Aging of Iron," *Stahl und Eisen*, January 2 and 9, 1919.

A. V. de Forest, "Some Experiments on the Plastic Elongation of Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part II, p. 455 (1916).

F. C. Lea and O. H. Crowther, "The Change of the Modulus of Elasticity and of Other Properties of Metals with Temperature," *Engineering*, Vol. 98, October 16, 1914, p. 487.

C. Bach, "Elastizität und Festigkeit," p. 174, Julius Springer, Berlin (1920).

300 F. (150 C.) and higher show numerous irregularities; this is most prominent at 400 F. (205 C.). These are believed to be due to the intermittent plastic deformation and age-hardening of the specimen during the test. The first deformation causes aging and concomitant hardening of the portion that has deformed. The specimen can then support a greater load than before and the curve rises until a load is reached that is sufficient to produce deformation at some other point. These deformations take place rapidly and the load drops off momentarily because of the inertia of the moving parts of the testing machine. The result is a series of "yield points" until the specimen finally breaks.

Under the test conditions described, the blue-brittle characteristics are most pronounced at 400 F. (205 C.). and so that testing temperature was selected for determining the properties in the blue-brittle range. The "blue-brittle" test, as the term is used here, consists in determining the tensile properties at room temperature and at 400 F. (205 C.). The differences between the tensile strength at room temperature and that at 400 F. (205 C.) and the roughness of the stress-strain curve are measures of the degree of blue brittleness.

Up to the present time there has been no ferrous material with physical properties completely stabilized against changes due to aging; therefore, it has been impossible for scientific workers to determine the effect of temperature on the tensile strength of such metals when they were free from blue brittleness. The development of a completely stabilized mild steel sheet material for deep-drawing purposes[1] offers an opportunity for such a study.

The dashed line in Fig. 3 shows the results of tension tests at elevated temperature on the stabilized steel sheet and it will be seen that there is no increase in strength above that at room temperature; as a matter of fact, the strength decreases continuously with rising temperature in the same manner as many non-ferrous materials, which, as is well known, are free from the objectionable phenomena of blue brittleness and instability. The stress-strain curves of this stabilized material tested at various testing temperatures are shown in the lower part of Fig. 4, and it will be observed that they are quite free from irregularities at all temperatures.

CORRELATION OF BLUE-BRITTLE TEST WITH STABILITY OF PHYSICAL PROPERTIES AND TENDENCY TO STRETCHER STRAIN

In studying the stability of the physical properties, the conditions that represent the variables encountered in the manufacture and use of deep-drawing sheets and those changes in properties which would affect the drawing qualities are of most interest, especially in the development of a material that does not stretcher strain within, say, six months or longer at room temperature. So, strips were cold rolled 2 per cent on an experimental cold mill and held 3 hours at the temperature of boiling water (which other tests have shown is about equivalent to 6 months at room temperature) and then tested in tension to determine the change in ductility, tensile strength, and the return of a sharp yield point and yield-point elongation. The latter two have been shown to be related very closely to stretcher straining.[1] In this study, commercial stability of physical properties against changes due to aging was interpreted as no increase in tensile strength, no loss in ductility measured by the elongation in the tension test, and no return of stretcher straining after holding three hours at 212 F. (100 C.). The amount of elongation at the yield point, as shown by an accurate stress-strain curve, can be used as a measure of the tendency to stretcher strain.

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[1] *The Iron Age,* July 13, 1933, p. 27.

TABLE I.—BLUE-BRITTLE AND AGING TESTS ON TEN MATERIALS WITH VARYING DEGREES OF STABILITY.[a]

Material	Blue-Brittle Tests			Aging Tests					
	Tensile Strength, lb. per sq. in.			Tensile Strength, lb. per sq. in.			Elongation in 2 in., per cent		
	At Room Temperature	At 400 F. (205 C.)	Difference, 400 F. (205 C.) to Room Temperature	Fresh	3 Hours at 212 F. (100 C.)	Difference	Fresh	3 Hours at 212 F. (100 C.)	Difference
No. 1	57 575	71 150	+13 575	60 300	64 600	+4300	24.25	17.25	−7.00
No. 2	47 575	58 200	+10 625	49 100	53 700	+4600	31.75	24.50	−7.25
No. 3	47 950	57 050	+9 100	49 700	53 125	+3425	31.25	22.75	−8.50
No. 4	41 000	42 075	+1 075	41 375	42 250	+875	40.00	37.25	−2.75
No. 5	44 390	44 200	−190	46 100	46 425	+325	38.75	37.25	−1.50
No. 6	43 450	42 800	−650	44 275	44 275	0	35.75	33.25	−2.50
No. 7	42 800	41 425	−1 375	42 650	42 675	+25	39.50	39.50	0
No. 8	59 200	53 400	−5 300	61 975	61 225	−750	29.75	30.25	+0.50
No. 9	45 300	37 725	−7 575	46 375	46 075	−300	39.00	38.75	−0.25
No. 10	57 650	48 225	−9 425	61 725	60 950	−775	32.50	32.50	0

[a] Each value shown is an average of two determinations. These two determinations differed probably from chance causes since the specimens were carefully selected to be alike as far as anyone knew. The average, maximum, and minimum differences between duplicate specimens were as follows:

	Blue-Brittle Tests		Aging Tests			
	Room Temperature	400 F. (205 C.)	Tensile Strength		Elongation	
			Fresh	Aged	Fresh	Aged
Average difference	397	456	415	680	0.78	1.28
Maximum difference	1250	1800	1300	1650	1.5	2.5
Minimum difference	20	50	50	50	0	0

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[1] R. L. Kenyon and R. S. Burns, "Autographic Stress-Strain Curves of Deep Drawing Sheets," *Transactions*, Am. Soc. Steel Treating, July, 1933.

Small changes in actual yield-point values in pounds per square inch due to aging are difficult to determine consistently because the same method of measurement cannot always be used. The freshly cold-rolled material does not have a sharp transition point, and it is necessary to adopt some arbitrary amount of strain for determining the yield stress.[1] If aging causes the return of a sharp yield point, the yield-point value is taken at this sharp transition. The "0.5 per cent yield stress" is then not significant. For this reason, changes in yield stress due to aging may fluctuate considerably and are not considered here.

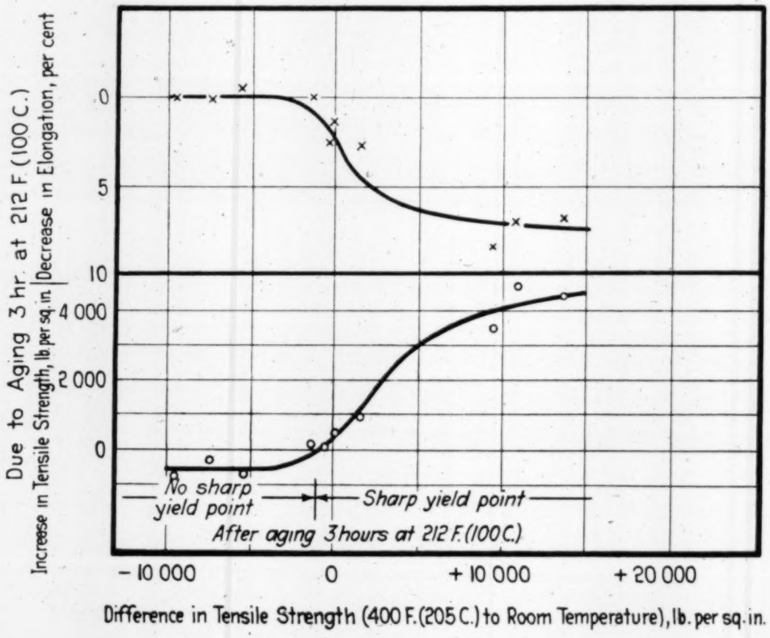

FIG. 5.—Relationship Between Changes in Tensile Strength and Percentage Elongation Due to Aging 3 hr. at 212 F. (100 C.) After Cold Rolling 1 per cent, and the Difference Between Tensile Strength at 400 F. (205 C.) and Room Temperature.

In the development of a stabilized steel for deep-drawing purposes, materials with varying degrees of stability have been tested. They range from mild steel to the stabilized material. In order to determine a definite relationship between blue brittleness and stability of physical properties, it was necessary to make both blue-brittle and aging tests on materials covering this range.

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[1] R. L. Kenyon and R. S. Burns, "Autographic Stress-Strain Curves of Deep Drawing Sheets," *Transactions*, Am. Soc. Steel Treating, July, 1933.

Table I gives the results of the blue-brittle and aging tests of a few of the materials tested. They are listed in order of increasing difference in tensile strength between 400 F. (205 C.) and room temperature, which places them in sequence according to decreasing blue brittleness. Figure 5 shows the change in tensile strength and

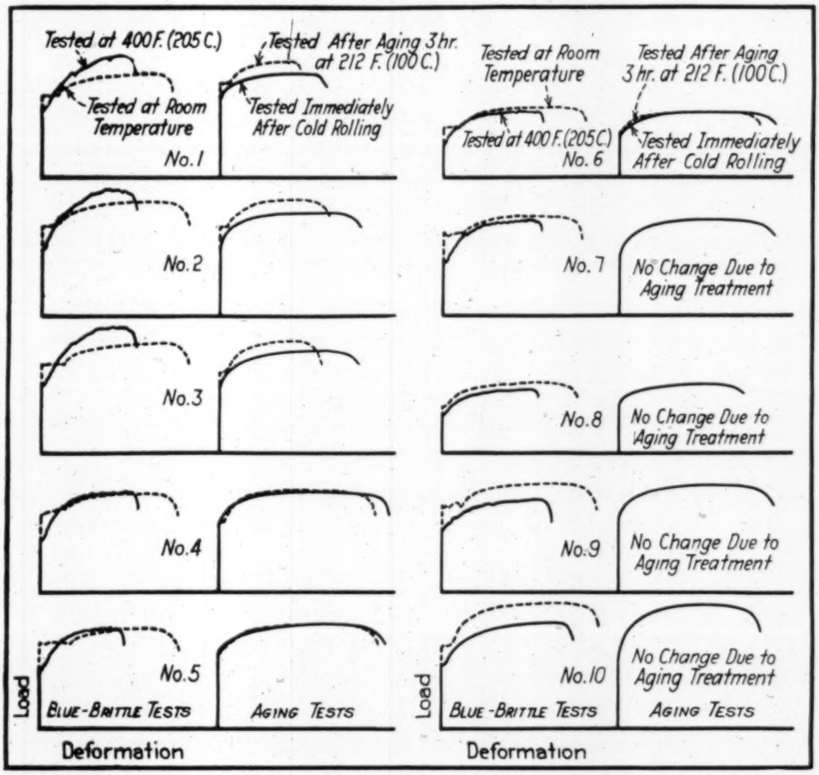

FIG. 6.—Comparison of Load Deformation Curves Obtained in the Blue-Brittle Test with Those in the Aging Test on the 10 Materials Given in Table I.

The numbers refer to the materials and are in the same order as in Table I. For the blue-brittle test the curves taken at 400 F. (205 C.) are to be compared with those at room temperature. For the aging test, the curves for the aged specimens are to be compared with those for the "fresh" specimens which were tested immediately after cold rolling.

percentage elongation resulting from aging 3 hours at 212 F. (100 C.) plotted against one measure of the blue brittleness (difference between tensile strength at 400 F. (205 C.) and at room temperature). Representative load-deformation curves for the tests tabulated in Table I are shown in Fig. 6. The results of the test used to measure blue brittleness are to the left of the number designating the material, while the load-deformation curves obtained in the aging test are to the right. The materials are arranged in the same order as in the table and it will be seen that the roughness of the curves for the tension tests at 400 F. (205 C.) decreases from material No. 1 to No. 10. The changes due to aging likewise decrease and materials Nos. 7, 8, 9, and 10 show no differences in their load-deformation curves before and after aging nor are there any significant changes in the numerical values given in Table I.

A study of these results shows that as long as the tensile strength at 400 F. (205 C.) is not greater than at room temperature, the tensile strength and ductility of material cold-rolled 2 per cent are practically stable against changes due to aging. The fact that the sharp yield point returns between −650 and −1375 lb. per sq. in. difference in tensile strength (400 F. (205 C.) to room temperature) is evidence that there is a discontinuity or change in the relationship and this coincides with the discontinuity in the numerical data as shown in Fig. 5. It will be noted from Fig. 6 that not only does the material exhibit a sharp yield point, after aging under the conditions stated, when the difference in the blue-brittle test is less than −1375 lb. per sq. in., but the amount of yield-point elongation increases with increasing blue brittleness.

In order to use the difference in tensile strength (400 F. (205 C.) to room temperature) as a measure of the stability of the drawing properties of sheet steel, it is necessary to consider both return of stretcher straining, as indicated by the yield-point elongation and the constancy of the tensile strength and percentage of elongation. A minimum difference in tensile strength (400 F. (205 C.) to room temperature) may then be adopted, which will insure any desired degree of stability in drawing properties against changes due to aging. A large number of various materials have been tested according to this procedure and in all cases it has been possible to predict whether the material would age after cold rolling.

AGING EMBRITTLEMENT OF 4 TO 6 PER CENT CHROMIUM STEEL

By H. M. Wilten[1] and E. S. Dixon[1]

Synopsis

Low-carbon steel containing in addition 4 to 6 per cent chromium is being extensively used in the oil refineries in the form of heater tubes. The steel of this analysis is at times subject to embrittlement after exposure to operating temperature (approximate range of 900 F., 480 C.). While being cleaned of coke deposit a small percentage of tubes crack. This cracking was found to be due to shock stresses on embrittled tubes.

The nature of this embrittlement has been investigated. It has been found that it is similar to duralumin type aging, when aging is carried out at room temperature. It is the thought of the authors that aging of this analysis is not due to actual precipitation of some element and some evidence is brought out to support their view. It is also postulated that this type of aging is similar to temper brittleness, where loss of impact occurs without a corresponding considerable increase in hardness and tensile strength commonly associated with aging of precipitation type.

Introduction

The needs of the oil industry with reference to the use of metals in equipment subjected to high pressures and temperatures have been recently clearly brought out(1).[2] As a result of severe conditions under which the metals in a modern oil refinery operate, the use of alloy steels was made imperative. At first, steels of stainless steel type were used. Later, steels of austenitic type, 18 per cent chromium, 8 per cent nickel, were tried, and, finally, a 4 to 6 per cent chromium steel was developed.

This latter steel is meeting with marked success at the present time, and although its physical properties are generally established, still some of its important features exhibited during actual service and the properties resulting from such service are not generally known, particularly, embrittlement in service. It seemed appropriate, therefore, that these service properties should be made known to the industry in general, with the hope that this additional information would lead to better understanding of the use of this steel, and to stimulate research to eliminate the undesirable features.

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[1] Metallurgist and Chief Metallurgist, respectively, The Texas Company, Port Arthur, Tex.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 77.

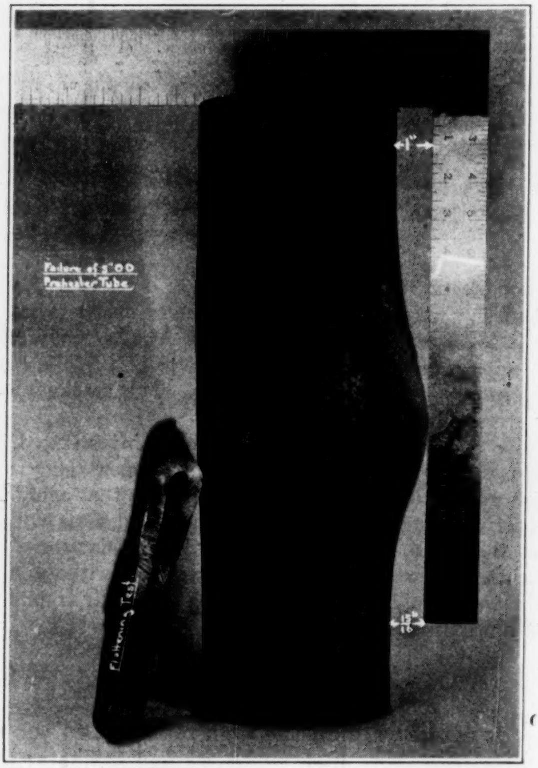
Failure of 5" OD
Preheater Tube
1"
Flattening Test
13/16"

FIG. 1.—Creep of Metal Resulting from Overheating of Heater Tube.

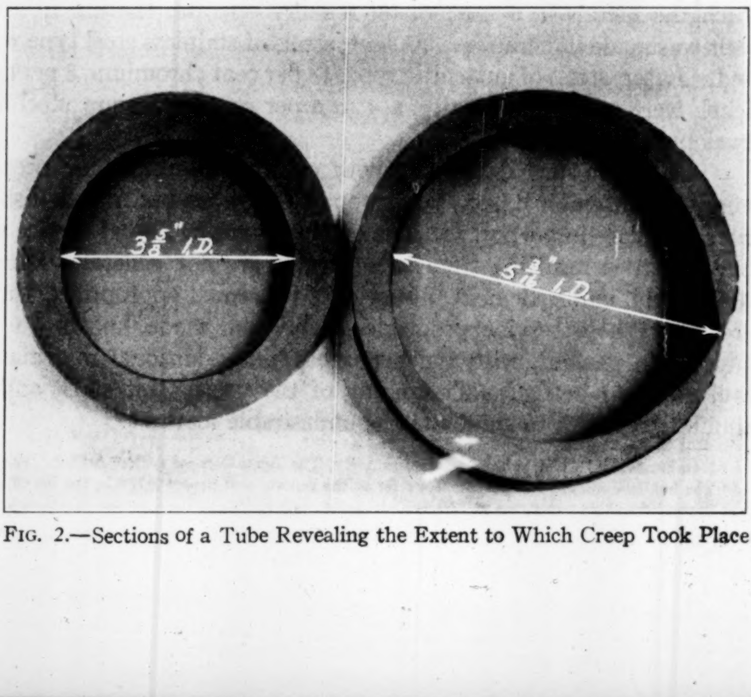
3 5/8" I.D.
5 3/16" I.D.

FIG. 2.—Sections of a Tube Revealing the Extent to Which Creep Took Place.

ANALYSIS

The chemical specifications under which this steel is commonly purchased are as follows:

Carbon, as specified, per cent	0.12 0.15 max. 0.20
Manganese, max., per cent	0.50
Silicon, max., per cent	0.50
Sulfur, max., per cent	0.04
Phosphorus, max., per cent	0.04
Chromium, per cent	4 to 6

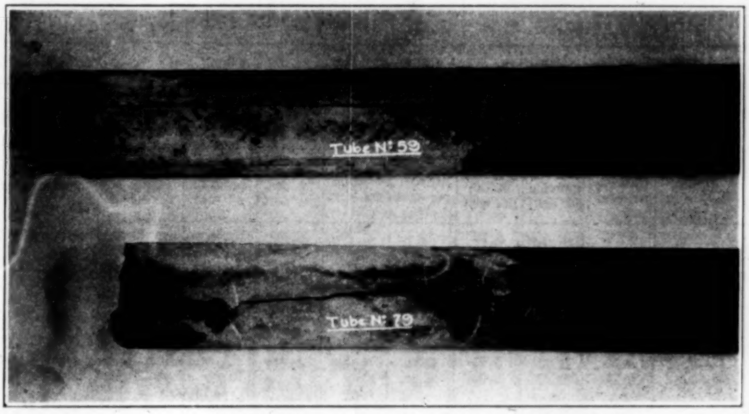

FIG. 3.—Cold Split—Revealing No Creep of Metal.

NORMAL SERVICE FAILURES

This type of steel when overheated swells to a considerable extent. Figure 1 shows the amount of swelling of a tube before actual rupture took place, and Fig. 2 shows sections through another tube revealing the extent to which creep took place. This failure represents typical normal failure. It occurs generally from severe coking of the oil, which allows the metal temperatures to increase rapidly above normal.

ABNORMAL FAILURES OF 4 TO 6 PER CENT CHROMIUM TUBES

Within the last two years and especially during the winter months, several 4 to 6 per cent chromium heater tubes split during the inspection period. The cracking unit makes a run of several hundred hours—until forced to shut down, usually due to coke formation in the battery. During the down period, the tubes are cleaned of coke and inspected before starting another run. It was during this inspection period that the tubes were found split (Fig. 3).

TABLE I.—SERVICE CONDITIONS OF FAILED TUBES.

NOTE.—Failures of tubes occurred after batteries were down several days. Failures occurred at atmospheric pressure.

Tube	Service Life, months	Atmospheric Temperature at Cleaning		Maximum Operating Temperature of Metal		Operating Pressure of Oil, lb.
		deg. Fahr.	deg. Cent.	deg. Fahr.	deg. Cent.	
A	6	40	4.5	1230	665	730
B	29	40	4.5	1190	645	740
C	32	60	1.5	960	515	940
D	29	27	−3	1070	575	625
E	29	27	−3	1100	595	665
F	26	30	−1	1125	605	685
G	30	30	−1	1050	565	450
H	30	30	−1	1190	645	740
I	26	42	5.5	1050	565	450

TABLE II.—CHEMICAL ANALYSIS AND LOCATION OF SPLITS.

Tube	Carbon, per cent	Manganese, per cent	Chromium, per cent	Silicon, per cent	Sulfur, per cent	Phosphorus, per cent	Location, Nature and Length of Split
A	0.19	....	5.26	....	....	....	10 in. above bottom terminal fitting 2 ft., 10 in. long
B	0.17	....	5.40	....	....	....	Right above bottom terminal 2 ft., 9 in. long
C	0.20	....	5.15	....	....	....	Top portion of the tube enclosed by the brick
D	0.21	....	5.49	....	....	....	Very close to the top
E	0.16	....	5.17	....	....	....	Very close to the top
F	0.20	0.32	5.21	0.31	0.024	0.019	Top and middle section over 5 ft. long
G	0.21	0.29	5.06	....	....	....	Right above bottom terminal fitting 13 in. long
H	0.22	0.29	5.27	....	....	....	From top terminal fitting 19 ft., 6 in. long
I	0.20	....	4.99	....	....	....	Beneath the bottom furnace sheet blocks

TABLE III.—TENSILE PROPERTIES OF FAILED TUBES.

Tube	Split Section	Top Section			Middle Section			Bottom Section		
		Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
A	Bottom	......	....	....	......	....	....	......	....	....
B	Bottom	......	....	....	......	....	....	......	....	....
C	Top	62 800	32.5	88.8	61 300	35.0	79.3	77 200	27.5	77.4
D	Top	......	....	....	......	....	....	......	....	....
E	Top	......	....	....	......	....	....	......	....	....
F	Top and middle	......	....	....	......	....	....	......	....	....
G	Bottom	69 200	30.5	76.9	......	....	....	82 600	26.0	74.4
H	Top	72 800	28.5	88.8	64 800	32.5	81.3	67 700	32.5	75.6
I	Bottom	......	....	....	67 800	37.0	....	......	....	....

Correlation Data of Abnormally Failed Tubes:

In order to correlate and to study their relative importance, all the factors which might relate to these failures have been collected in Tables I to IV, for nine cases of cracked tubes. These constitute failures occurring among a large group of tubes purchased on the same order.

A study of service conditions showed:

1. Practically all tubes had a service life of over two years.
2. Most of the tubes failed at a relatively cold atmospheric temperature.
3. There was no preferential location of failures with respect to oil temperatures and pressures.

TABLE IV.—CHARPY IMPACT PROPERTIES OF FAILED TUBES.

NOTE 1.—The values are averages of at least three tests closely agreeing with each other.
NOTE 2.—Notches were cut on the tube thickness surface between the inside and the outside surfaces of the tubes.
NOTE 3.—These specimens were 60 mm. long.

Tube	Split Section	Top Section			Middle Section			Bottom Section		
		Standard Specimen, Keyhole Notch, ft-lb.	Alternate Specimen, V Notch, ft-lb.	Brinell Hardness	Standard Specimen, Keyhole Notch, ft-lb.	Alternate Specimen, V Notch, ft-lb.	Brinell Hardness	Standard Specimen, Keyhole Notch, ft-lb.	Alternate Specimen, V Notch, ft-lb.	Brinell Hardness
A	Bottom	51.2	18.7	121	46.9	16.3	....	50.1	18.0	116
B	Bottom	16.0	17.0	137	58.0	81.0	128	37.0	14.7	131
C	Top	41.0	15.0	116	42.5	16.5	121	38.0	31.0	143
D	Top	28.0	16.8	137	42.3	54.5	146	37.5	36.6	187
E	Top	....	....	128	....	....	....	....	....	....
F	Top and middle	....	....	156	....	....	....	....	....	....
G	Bottom	51.6	95.4	146	42.5	71.8	149	37.0	16.0	126
H	Top	36.0	20.0	131	....	....	....	38.3	44.8	167
I	Bottom	....	....	....	....	....	....	....	....	116

A study of physical properties showed:

1. Tensile properties are uniformly good in all sections of tubing.
2. Impact properties of V-notch specimens correlated with the failures in all cases. The material in the failed portions is notch-brittle.

For the sake of comparison, impact properties on three new 4 to 6 per cent chromium tubes are given in the following table:

	CHARPY IMPACT OF NEW TUBES, FT-LB.		
TUBE	STANDARD CHARPY SPECIMEN, KEYHOLE NOTCH	ALTERNATE CHARPY SPECIMEN, SPECIAL V NOTCH	BRINELL HARDNESS
K	54.5	126	160
L	55.5	113	150
M	41.5	64	152

In addition to the impact tests, the shape and nature of the fractures of these tubes also indicate a state of brittleness in the metal, with no flow of metal taking place prior to fracture.

The cause of failure of these tubes was found on investigation to be due to cleaning (knocking) of tubes when they were at a low temperature and accordingly had low resistance to shock stresses. It was also found, experimentally, that the internal stress was due principally to the cleaning operation. The fact that the metal suffers a drop in impact value as a result of prolonged exposure to elevated temperatures is of great interest. Low resistance to impact manifests itself in these tubes only at ordinary temperatures. As soon as the temperature is again raised, even to 212 F. (100 C.), practically all of the impact value of the metal is recovered.

TABLE V.—CHEMICAL ANALYSIS OF STEELS TESTED.

Designation	Type of Steel	Carbon, per cent	Chromium, per cent	Nickel, per cent	Silicon, per cent	Molybdenum, per cent	Tungsten, per cent	Titanium, per cent	Aluminum, per cent
CS 1	Plain carbon	0.12	....	....	....	....	....	....	....
CS 2	Plain carbon	Low	....	....	....	....	....	....	....
CS 3	Plain carbon	0.14	....	....	....	....	....	....	....
Cr 1	Plain chromium	0.19	5.04	....	0.03	....	....	....	....
Cr 2	Plain chromium	0.14	5.18	....	0.27	....	....	....	....
Cr 3	Plain chromium	0.07	5.28	....	0.39	....	....	....	....
Cr 4	Plain chromium	0.15	4.32	....	0.13	....	....	....	....
Cr Si 1	Chromium-silicon	0.14	5.21	....	1.76	....	....	....	....
Cr Si 2	Chromium-silicon	0.07	5.27	....	1.84	....	....	....	....
Cr Mo 1	Chromium-molybdenum	0.08	5.30	....	0.98	1.02	....	....	....
Cr Mo 2	Chromium-molybdenum	0.10	5.14	....	....	2.10	....	....	....
Cr Mo 3	Chromium-molybdenum	0.12	5.15	....	....	0.60	....	....	....
Cr Mo 4	Chromium-molybdenum	0.185	4.24	....	....	0.55	....	....	....
Cr Mo 5	Chromium-molybdenum	0.07	5.57	....	0.26	0.44	....	....	....
Cr W 1	Chromium-tungsten	0.11	5.34	....	0.48	....	0.90	....	....
Cr Ti 1	Chromium-titanium	0.16	4.23	....	0.38	....	....	0.27	....
Cr Al 1	Chromium-aluminum	0.195	4.62	....	0.40	....	....	....	0.93

EXPERIMENTS

Aside from corrosion resistance, the physical stability at all temperatures of service is the most important property of metal to be considered in oil refinery use.

Just as resistance to corrosion is usually determined by exposing corrosion specimens of different steels to the corrosive influence of oils in actual refining units under various conditions of temperature and pressure, so in like manner the same methods were used to determine the stability of physical properties of the tested alloys. Service conditions were simulated not only by exposing the specimens to various temperatures, but also by subjecting them to alternate heating and cooling cycles. Various standard methods for determining age-hardening and temper-brittleness were also used. These are described

TABLE VI.—TENSILE AND IMPACT PROPERTIES.

Type of Steel	Tensile Strength, lb. per sq. in.				Elongation in 2 in., per cent				Reduction of Area, per cent				Izod Impact Value, ft-lb.				Brinell Hardness			
	Original	670 F. (355 C.)	750 F. (400 C.)	815 F. (475 C.)	Original	670 F. (355 C.)	750 F. (400 C.)	815 F. (475 C.)	Original	670 F. (355 C.)	750 F. (400 C.)	815 F. (475 C.)	Original	670 F. (355 C.)	750 F. (400 C.)	815 F. (475 C.)	Original	670 F. (355 C.)	750 F. (400 C.)	815 F. (475 C.)
CS 1, plain carbon	44 300	44 250	43 900	45 500	47	44	45.5	43	72.7	72.9	74.5	68.2	30.5	51	49.3	50	76	78	80	78
Cr 1, 4 to 6 per cent chromium	63 700	66 700	64 000	66 625	38.5	38	40.5	39.8	83.2	82.3	83.2	80.8	115.8	115.8	115.8	64.3	107	117	114	117

TABLE VII.—TENSILE PROPERTIES OF ANNEALED AND EXPOSED SPECIMENS.

Type of Stee	Tensile Strength, lb. per sq. in.				Elongation in 2 in., per cent				Reduction of Area, per cent			
	Original	670 F. (355 C.)	900 F. (480 C.)	1240 F. (670 C.)	Original	670 F. (355 C.)	900 F. (480 C.)	1240 F. (670 C.)	Original	670 F. (355 C.)	900 F. (480 C.)	1240 F. (670 C.)
CS 1, carbon	60 125	61 125	60 750	52 700	33	32.5	34	39	60.8	55.7	56.5	66.5
Cr 1, 4 to 6 per cent chromium	63 500	64 000	75 750	62 800	38	35	32.5	40	82.6	82.1	78.0	83.2
Cr 2, 4 to 6 per cent chromium	73 250	......	73 875	......	29	.....	29	..	69	.....	69	.....
Cr 3, 4 to 6 per cent chromium	61 375	64 500	62 750	60 000	35	35.5	36	40	76.5	74.3	75	78.1
Cr Mo 1, 4 to 6 per cent chromium + silicon + molybdenum	72 500	75 500	......	70 350	32.5	32.5	....	33	88.4	63.3	....	67.7
Cr Mo 2, 4 to 6 per cent chromium + molybdenum	74 050	84 350	......	68 600	30	28	....	33	68.5	63	....	71.9
Cr Si 1, 4 to 6 per cent chromium + silicon	88 150	87 750	89 000	84 500	30	29	30	30	60.8	62.1	62.7	66.8
Cr Si 2, 4 to 6 per cent chromium + silicon	76 200	79 000	......	75 700	32.5	33	....	35	67.6	66.1	....	70.1

TABLE VIII.—TENSILE STRENGTH PROPERTIES OF ANNEALED SPECIMENS.

Type of Steel	Tensile Strength, lb. per sq. in.					Elongation in 2 in., per cent					Reduction of Area, per cent				
	Original	520 F. (270 C.)	670 F. (355 C.)	750 F. (400 C.)	900 F. (480 C.)	Original	520 F. (270 C.)	670 F. (355 C.)	750 F. (400 C.)	900 F. (480 C.)	Original	520 F. (270 C.)	670 F. (355 C.)	750 F. (400 C.)	900 F. (480 C.)
CS 2, plain carbon	56 350	56 000	56 000	55 300	55 250	34.5	35.0	36.0	35.0	35.0	55.5	56.8	58.6	57.0	56.0
Cr 1, 4 to 6 per cent chromium	66 300	67 000	67 000	65 000	64 500	38.0	39.0	38.0	39.0	39.5	82.5	83.0	82.0	63.0	82.5
Cr Mo 3, 4 to 6 per cent chromium + molybdenum	65 600	......	64 250	64 500	63 750	37.0	....	36.0	39.0	37.5	77.0	....	77.0	77.0	76.7
Cr W 1, 4 to 6 per cent chromium + tungsten	71 500	71 600	72 350	73 000	71 000	37.0	36.5	35.0	35.5	36.5	78.0	77.5	78.0	77.5	77.0

more fully in connection with each of the tests reported upon. For comparative purposes, carbon steels as well as 4 to 6 per cent chromium steels containing additional elements were tested at the same time. All of the steels were annealed prior to testing. The chemical analyses of the steels tested are shown in Table V. The values for impact test data represent averages of at least three determinations unless otherwise stated. The impact data were not averaged unless the values did not differ by more than 20 per cent.

TABLE IX.—IMPACT PROPERTIES AND BRINELL HARDNESS.

Type of Steel	Izod Impact Values, ft-lb.				Brinell Hardness, 1000-kg. load		
	Original	670 F. (355 C.)	900 F. (480 C.)	1240 F. (670 C.)	Original	900 F. (480 C.)	1240 F. (670 C.)
ANNEALED SPECIMENS							
CS 1, carbon	3.2	6.3	6.2	9.5	101	101	...
Cr 1, 4 to 6 per cent chromium	120+	120+	120+	120+	114	107	104
Cr 2, 4 to 6 per cent chromium	42.2	46.5	2.5	46.0	125	129	...
Cr 3, 4 to 6 per cent chromium	57.7	69	14.5	69	114	107	104
Cr Mo 1, 4 to 6 per cent chromium + silicon + molybdenum	73.3	78	63.3	13.0	129	129	121
Cr Mo 2, 4 to 6 per cent chromium + molybdenum	7.6	13.5	6.5	10.5	121	148	121
Cr Si 1, 4 to 6 per cent chromium + silicon	1.75	4	1.25	3	154	159	148
Cr Si 2, 4 to 6 per cent chromium + silicon	2.3	5	1.5	2.25	...	148	...

TABLE X.—IMPACT PROPERTIES AND BRINELL HARDNESS.

Type of Steel	Charpy Impact Values, ft-lb.					Brinell Hardness, 1000-kg. load				
	Original	520 F. (270 C.)	670 F. (355 C.)	750 F. (400 C.)	900 F. (480 C.)	Original	520 F. (270 C.)	670 F. (355 C.)	750 F. (420 C.)	900 F. (480 C.)
ANNEALED SPECIMENS										
CS 2, plain carbon	20.1	20.1	23.0	21.4	21.4	107	104	104	104	114
Cr 1, 4 to 6 per cent chromium	71.2	77.3	60.5	54.5	48.7	121	154	...	154	154
Cr Mo 3, 4 to 6 per cent chromium + molybdenum	54.9	53.1	71.5	56.8	54.5	121	121	115	114	121
Cr W 1, 4 to 6 per cent chromium + tungsten	53.6	60.4	59.0	60.8	49.6	129	134	136	134	133

Stability Tests:

Test No. 1.—In this test, annealed specimens were exposed at temperatures of 670, 750 and 815 F. (355, 400 and 435 C.), for 712, 457 and 940 hr., respectively. The tensile and impact properties before and after exposure are shown in Table VI and Fig. 4.

Test No. 2.—In this test, annealed specimens were exposed to temperatures as follows:

(*a*) 670 F. (355 C.) for 3166 hr.
(*b*) 780 F. (415 C.) for 3166 hr.
(*c*) 900 F. (480 C.) for 3423 hr.
(*d*) 1240 F. (670 C.) for 3166 hr.

The tensile and impact properties before and after exposure are shown in Tables VII and IX and Fig. 5.

Test No. 3.—Annealed specimens were exposed to temperatures as follows:

(*a*) 520 F. (270 C.) for 1260 hr.
(*b*) 670 F. (355 C.) for 2051 hr.
(*c*) 750 F. (400 C.) for 2051 hr.
(*d*) 900 F. (480 C.) for 1250 hr.

Tensile and impact properties are shown in Tables VIII and X and Fig. 6.

Test No. 4.—Annealed specimens were exposed to temperatures as follows:

(*a*) 750 F. (400 C.) for 2658 hr.
(*b*) 900 F. (480 C.) for 2559 hr.

Tensile and impact properties are shown in Tables XI and XII.

Temper Brittleness Tests:

Temper brittleness tests were made by the method recommended by Greaves and Jones(2). In this method, duplicate samples of the steels to be examined are hardened in water from 1650 F. (890 C.) and tempered at 1200 F. (650 C.); one sample is cooled in water after tempering and the other is cooled slowly in the furnace. Susceptibility to temper brittleness may be taken as the ratio of the impact figure of the sample rapidly cooled to that of the sample cooled slowly after tempering. The higher the ratio, the more brittle the material. In these tests, alternate Charpy specimens (V-notch, 0.079 in. in depth) were used unless otherwise stated. The impact properties obtained are shown in Table XIII.

GENERAL DISCUSSION

The stability tests show that resistance to impact of 4 to 6 per cent chromium steel is at times lowered when the steel is heated at 900 F. (480 C.) for a considerable interval of time. The susceptibility ratio of the steel to temper brittleness is also high.

The embrittlement referred to is usually ascribed to "temper brittleness" of the metal(3). This type of brittleness has been commonly associated with steels of S.A.E. 3100 and 3200 series when they are cooled slowly from the tempering operation.

In a group of impact test specimens taken either from the failed tubes or from experimental specimens subjected to prolonged heating, it has been our experience that individual specimens may show very erratic values. It appears that the steel is extremely sensitive to cooling from the tempering operation. After further studying this phenomenon, the authors were particularly impressed by the fact that prolonged heating at, say, 900 F. (480 C.) will produce embrittlement even though the cooling from that temperature may be fairly rapid. The time factor, whether due to the prolonged exposure to temperature or to the rate of cooling, became of especial significance when this point was studied in greater detail. Consideration of this time factor led to speculation as to whether the temper brittleness of 4 to 6 per cent chromium steel might not be a type of aging phenomenon. The fact that temper brittleness may also be a type of aging has also occurred to French(5).

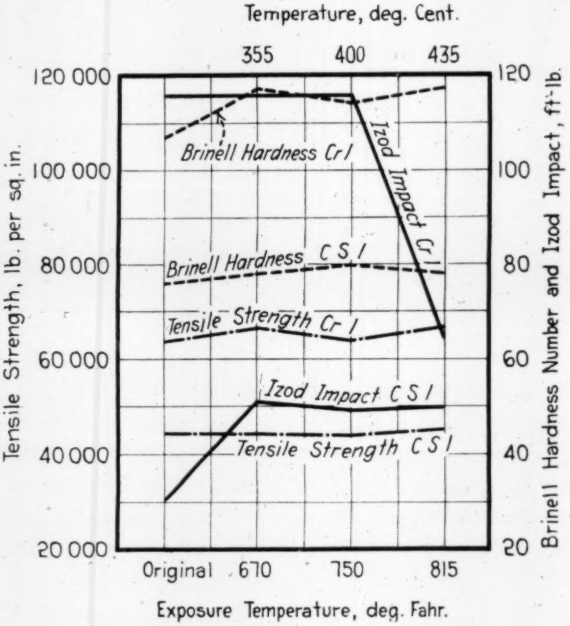

FIG. 4.—Change in Physical Properties of Carbon and 4 to 6 per cent Chromium Steels as a Result of Exposure of Steels to Elevated Temperatures.

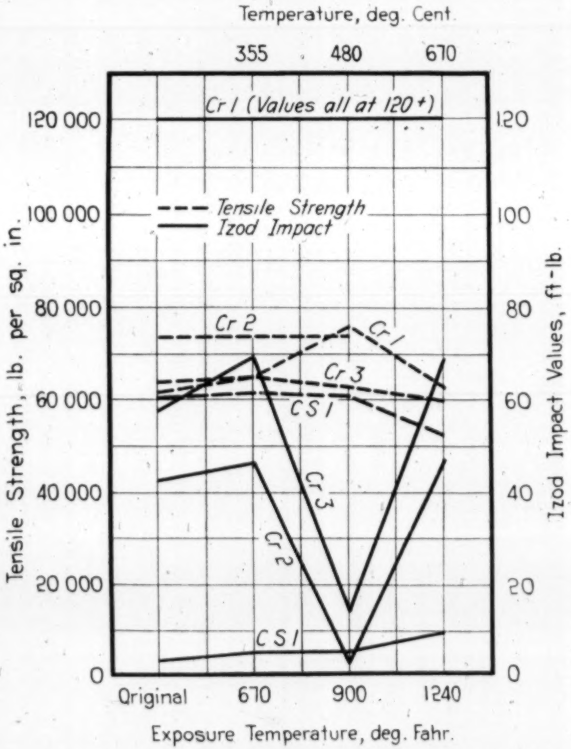

Fig. 5.—Change in Physical Properties of Carbon and 4 to 6 per cent Chromium Steels as a Result of Exposure of Steels to Elevated Temperatures.

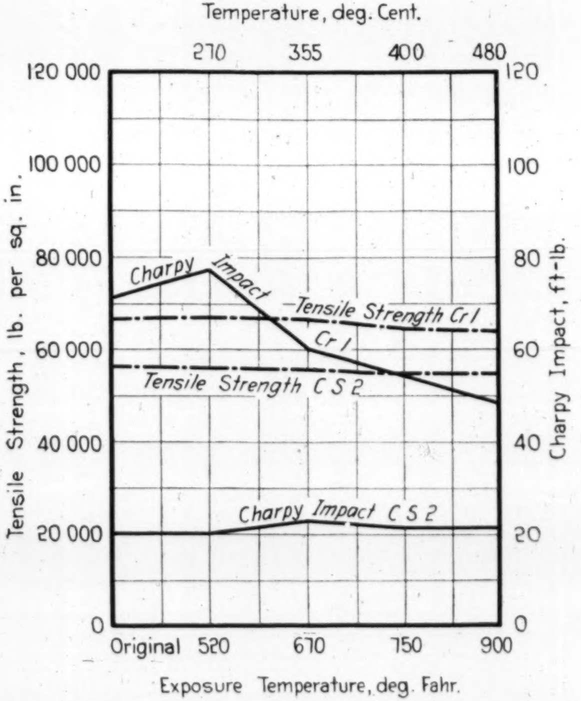

Fig. 6.—Change in Physical Properties of Carbon and 4 to 6 per cent Chromium Steels as a Result of Exposure of Steels to Elevated Temperatures.

The cause of temper brittleness has been ascribed by Rogers(6) to the separation of carbides from solution on slow cooling from the tempering operation. He showed that in typical heating and cooling curves in the heating cycle, there is absorption of heat between 755 to 935 F. (400 to 500 C.) which he thinks is due to resolution of carbides. This view is shared by Andrew and Dickie(7). This view has not been supported by Greaves and Jones(2) who trace temper brittleness to the presence of chromium oxide in the steel. Leaving these opinions at this time, without comment, let us study aging phenomena and their various explanations.

TABLE XI.—TENSILE PROPERTIES.

Type of Steel	Tensile Strength, lb. per sq. in.			Elongation in 2 in., per cent			Reduction of Area, per cent		
	Original	750 F. (400 C.)	900 F. (480 C.)	Original	750 F. (400 C.)	900 F. (480 C.)	Original	750 F. (400 C.)	900 F. (480 C.)
CS 3, plain carbon	50 150	49 150	47 850	42	44	45	73.6	69.9	74.7
Cr 1, 4 to 6 per cent chromium	64 000	65 500	67 850	37	37.5	38	76.6	81.9	80.8
Cr 4, 4 to 6 per cent chromium	58 750	58 750	58 400	40	39.0	41	77.4	78.6	77.8
Cr Mo 4, 4 to 6 per cent chromium + molybdenum	70 850	70 650	70 150	37	37.0	37.5	78.5	78.1	78.1
Cr Mo 5, 4 to 6 per cent chromium + molybdenum	105 250	111 000	102 750	22	20.0	21	74.9	68.3	74.3
Cr Ti 1, 4 to 6 per cent chromium + titanium	66 000	67 850	66 500	39	37.5	37.5	79.4	79.6	78.7
Cr Al 1, 4 to 6 per cent chromium + aluminum	73 100	72 250	73 250	36	36.0	32.5	72.4	73.1	73.9

TABLE XII.—CHARPY IMPACT VALUES.

Type of Steel	Alternate Charpy Specimens, V Notch, ft-lb.			Standard Charpy Specimens, Keyhole Notch, ft-lb.			Brinell Hardness		
	Original	750 F. (400 C.)	900 F. (480 C.)	Original	750 F. (400 C.)	900 F. (480 C.)	Original	750 F. (400 C.)	900 F. (480 C.)
CS 3, plain carbon	149.4	151.4	152.4	59.0	62.3	63.2	85	85	84
Cr 1, 4 to 6 per cent chromium	142.5[a]	219.1	84.7[b]	54.9	62.0	64.3	110	110	109
Cr 4, 4 to 6 per cent chromium	144.9	132.7	137.1	67.8	58.0	53.5	102	96.3	96.3
Cr Mo 4, 4 to 6 per cent chromium + molybdenum	196.9	193.7	188.4	74.3	78.9	72.4	114	121	119
Cr Mo 5, 4 to 6 per cent chromium + molybdenum	156.5	154.5	158.7	67.8	66.2	66.9	218	227	214
Cr Ti 1, 4 to 6 per cent chromium + titanium	213.5	209.2	215	69.0	63.4	58.8	118	113	113
Cr Al 1, 4 to 6 per cent chromium + aluminum	119.0	80.3	40.1	46.2	43.4	36.2	126	124	123

[a] Results erratic; represents the average of 166.7, 183.3, 165.2, 54.7.
[b] Results erratic; represents the average of 24, 15.7, 80.6, 218.7.

Under aging[1] Mehl(8) groups three types of phenomena:

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[1] The authors prefer to use the word "aging" even though some writers are using and advocating the term "age-hardening"(4). It is felt that, first, aging adequately describes change of properties of steel as a result of time factor and temperature change, and, second, because in iron alloys, aging may not necessarily produce increase in hardness(5).

1. Blue brittleness of steel—diminished ductility at 550 to 750 F. (290 to 400 C.).

2. Aging of overstrained steel—change in properties on storage or reheating to moderate temperatures of cold-worked steel.

3. Duralumin type of aging produced by quenching a steel alloy below the A_1 point, subsequently followed by storage or heating to moderate temperatures.

The theory of the third type of aging, even though discovered in steel only a few years ago, is well developed. The conditions necessary for occurrence of this type of aging require that the alloy be obtainable at ordinary temperature in the state of a supersaturated solution. See Fig. 7, taken from Masing(9).

TABLE XIII.—TEMPER BRITTLENESS.

Type of Specimen	V-Notch Specimen					Keyhole-Notch Specimen				
	Quenched Specimen		Furnace-Cooled Specimen		Ratio	Quenched Specimen		Furnace-Cooled Specimen		Ratio
	Charpy Impact Values, ft-lb.	Brinell Hardness	Charpy Impact Values, ft-lb.	Brinell Hardness		Charpy Impact Values, ft-lb.	Brinell Hardness	Charpy Impact Values, ft-lb.	Brinell Hardness	
EXPERIMENT No. 5										
Plain carbon	106	115	155.3	109	0.69	....	..	....	..	....
Cr 1, 4 to 6 per cent chromium	162.3	192	38.1	197	4.26	....	..	....	..	....
A^a, 4 to 6 per cent chromium	119.4	189	25.6	202	4.67	....	..	....	..	....
D^a, 4 to 6 per cent chromium	103.8	223	24.3	212	4.25	....	..	....	..	....
Cr Mo 3, 4 to 6 per cent chromium + molybdenum	97.5	217	99.5	222	0.98	....	..	....	..	....
Cr W 1, 4 to 6 per cent chromium + tungsten	178.2	189	118.9	195	1.51	....	..	....	..	....
EXPERIMENT No. 6										
CS 3, plain carbon	143.5	105	183	82	0.78	58.5	95	63.6	85	0.92
Cr 1, 4 to 6 per cent chromium	172.8	185	102	159	1.7	69.3	178	31.2	187	2.22
Cr 4, 4 to 6 per cent chromium	111.1	170	110.2	174	1.0	47.4	166	51.3	163	0.92
Cr Mo 4, 4 to 6 per cent chromium + molybdenum	125	182	125	204	1.0	55.4	192	52.2	197	1.06
Cr Mo 5, 4 to 6 per cent chromium + molybdenum	169.3	179	170.6	175	1.0	73.1	163	74.4	166	0.98
Cr Ti 1, 4 to 6 per cent chromium + titanium	164.3	177	152.6	175	1.07	68.7	187	64.3	170	1.07
Cr Al 1, 4 to 6 per cent chromium + aluminum	134.9	178	117.3	179	1.15	57.6	184	30.7	190	1.88

[a] Specimens marked A and D were taken from Tubes A and D which failed in abnormal manner. See Table III.

The alloy is homogeneous at temperatures above t_2 and is also obtained in a homogeneous state at t_o, room temperature, when quenched from temperature t_2. This alloy will age-harden when heated to an intermediate temperature t_1. These conditions certainly indicate that this aging process is connected with the decomposition or "decay" of solid solution.

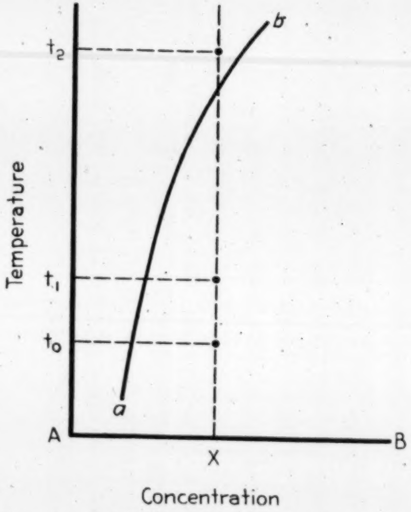

FIG. 7.—Solubility Conditions Necessary for Aging of Duralumin Type.

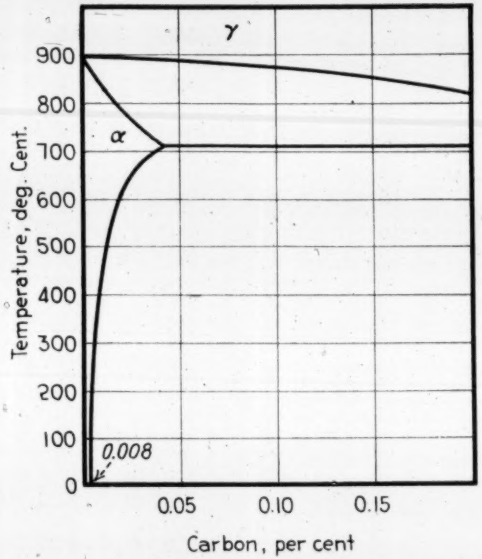

FIG. 8.—Solubility Diagram of Carbon in Alpha Iron.

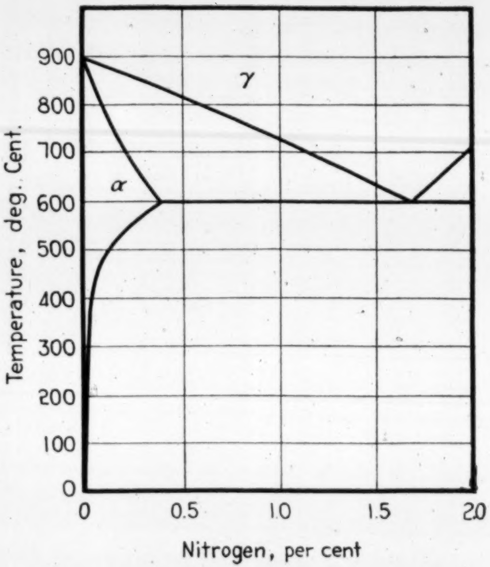

FIG. 9.—Solubility Diagram of Nitrogen in Alpha Iron.

This brief description of temper brittleness and aging phenomena indicates that, according to the consensus of opinion, the cause of both phenomena appears to be tied up with the change in solubility of some elements in alpha iron.

Consider the problem of embrittlement of 4 to 6 per cent chromium in service. It has already been stated that at 212 F. (100 C.) a large percentage of the impact value of embrittled metal is recovered. Table XIV presents actual figures obtained.

TABLE XIV.—CHARPY IMPACT VALUE OF 4 TO 6 PER CENT CHROMIUM STEEL FROM EMBRITTLED TUBES AT ROOM AND ELEVATED TEMPERATURES.

Identification	Charpy Impact Values, ft-lb. (Alternate Specimens, V Notch)				
	75 to 85 F. (24 to 29 C.)	100 F. (38 C.)	150 F. (66 C.)	200 to 212 F. (93 to 100 C.)	300 F. (150 C.)
40[a]	6.0	5.0	8.0	54.3	149.8
	2.3	4.1	10.2	18.6	....
42[a]	5.0	45.7	49.1	57.7	156.9
	2.3	4.1	16.0	142.5	....
D	45.3[d]	....	....	155	....
A[b]	16.3[d]	....	....	177	....
A[c]	18.0[d]	....	....	67.4	....
E	12.6[d]	....	....	170.2	....

[a] Individual values.
[b] Specimens taken from the middle portion of the tube.
[c] Specimens taken from the bottom portion of the tube.
[d] Average of at least two values.

TABLE XV.—BRINELL HARDNESS VALUES SHOWING ARTIFICIAL AGING OF 4 TO 6 PER CENT CHROMIUM STEEL.

Type of Steel	Quenching Temperature, 1300 F. (705 C.)		Quenching Temperature, 1400 F. (760 C.)		Quenching Temperature, 1500 F. (815 C.)	
	As Quenched	Aged[a]	As Quenched	Aged[a]	As Quenched	Aged[a]
CS 3, plain carbon	121	179	116	183	111	163
Cr 1, 4 to 6 per cent chromium[b]	116	118	111	127	229	241
Cr 4, 4 to 6 per cent chromium	111	116	116	121	207	207
Cr Mo 4, 4 to 6 per cent chromium + molybdenum	137	140	143	146	143	156
Cr Mo 5, 4 so 6 per cent chromium + molybdenum	179	179	156	159	149	152
Cr Ti 1, 4 to 6 per cent chromium + titanium	131	131	131	137	137	137
Cr Al 2, 4 to 6 per cent chromium + aluminum	137	149	143	149	149	152

[a] Aging consisted of storage of specimens for 30 days at room temperatures.
[b] The Ac_1 point in 4 to 6 per cent chromium steel 1470 F. (800 C.) and Ar_1 is 805 F. (430 C.), from Becket and Franks (10).

Figures 8 and 9, taken from Mehl(8), show the solubility of carbon and nitrogen in alpha iron at different temperatures. They show practically no change in solubility from room temperature to 212 F. (100 C.). It would, therefore, appear that specimens embrittled due to the third (duralumin) type of aging should not show much different impact value at 212 F. (100 C.). However, specimens in

Table XIV do show remarkable improvement in impact value, which in turn indicates that the embrittlement of 4 to 6 per cent chromium steel is not due to precipitation hardening.

This was confirmed by the following test, using the original method for aging outlined by Masing and Koch(12). The specimens were annealed and quenched from around the A_1 point, and aged:

It is to be noted from Table XV that, while carbon steel showed considerable increase in hardness after aging, the 4 to 6 per cent chromium steels did not. This is also illustrated in Fig. 10. It is to be further noted that steel Cr 1 which had previously been shown to have high susceptibility to temper brittleness as well as considerable drop in impact value after exposure to 900 F. (480 C.) did not age-harden, showing that 4 to 6 per cent chromium steel is not subject to precipitation hardening. It is of interest to note here that the same steels as shown in Table XV when quenched from 1110 F. (600 C.) (point of greatest solubility of nitrogen in steel) and aged, did not show any age-hardening, except carbon steel which showed some hardening. The same steels when quenched from 1400 and 1500 F. (760 and 815 C.) and aged for one hour respectively at 400, 600, 800, 1000 and 1200 F. (205, 315, 425, 540 and 650 C.) did not show any increase in hardness.

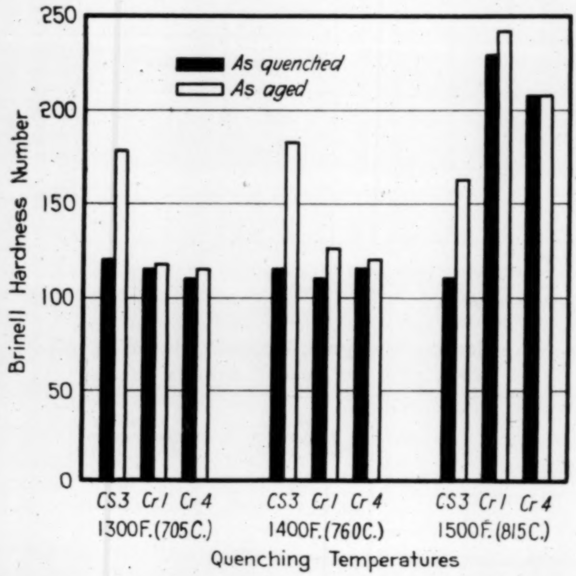

FIG. 10.—Change in Brinell Hardness of Steels After Aging Test.

In the paper by Mehl(8) referred to, he has shown the similarity of all three types of aging. If the authors' contention that during the embrittlement of 4 to 6 per cent chromium steel no precipitation of any constituent takes place, the embrittlement of 4 to 6 per cent chromium steel does not come within the scope of aging thus far described.

It has already been mentioned that several investigators postulated that temper brittleness of steels is also due to precipitation of an unknown constituent. In studying the fatigue resistance of temper-brittle steels, French(5) showed that fatigue limit of quenched temper-brittle steel is higher than that of slowly cooled steel, even though the tensile strength of the former is less than that of the latter. He very convincingly ascribes this to "slip interference produced by particle precipitation in the slip bands formed during repeated stressing."

He further states that "the precipitation accepted as the cause of temper brittleness may not be that which caused the strengthening under repeated stressing, since the former occurs at relatively high temperatures around 1100 F. (595 C.) at which diffusion can readily take place, while the latter is observed under stresses applied at atmospheric temperatures."

It is believed that the data brought out by French convincingly show that this is what occurs. He showed that nickel-silicon steel (temper brittleness susceptibility ratio of 5) showed persistent improvement on overstressing, both in a quenched state and when tempered at 675 F. (355 C.), but no strengthening was developed by overstressing samples tempered at 1200 F. (650 C.) either water quenched or furnace cooled.

The difference in behavior of steel with a susceptibility ratio of 5 when tempered from 675 and 1200 F. (355 and 650 C.) is rather significant in showing that the phenomenon causing the fatigue strengthening at a lower temperature may be different from the process occurring at a higher temperature.

The simple "dispersion" or precipitation theory of age-hardening of duralumin alloys first proposed by Merica, Walterberg and Scott(13) which, while accounting for aging of duralumin at high temperature (100 to 250 C., 212 to 480 F.) did not explain the anomalous behavior in electrical properties of duralumin when it is aged at ordinary temperatures. This question has been extensively treated by Merica in a very recent paper(11). He concludes that while the aging of duralumin at higher temperatures is unquestionably due to the precipitation of crystalline particles of $CuAl_2$, the age-hardening of duralumin at ordinary temperatures "may also occur in consequence of some structural alteration other than that of the precipitation of excess solute."

Is it not possible that temper brittleness in which alteration of impact properties occurs without increase in hardness or tensile strength is a result of structural alteration without actual precipitation of any constituent, since a specimen aged by actual precipitation of a hard constituent would become harder throughout the aggregate?

Our experiments on stability of 4 to 6 per cent chromium steels show that lower impact value, when it occurred, always was accompanied by no appreciable increase either in hardness or tensile properties. It is also worthwhile to note that there appears to be a correlation between the stability of the 4 to 6 per cent chromium steels and their susceptibility to temper brittleness. The metal in the abnormally failed tubes was temper brittle. The steel Cr 1 which showed lower impact value after exposure to a temperature of 900 F. (480 C.) was also temper brittle; 4 to 6 per cent chromium steel Cr 4 which showed no deterioration of impact properties after exposure was found not to be susceptible to temper brittleness. The same is true of 4 to 6 per cent chromium steels alloyed with molybdenum. The similarity of behavior of steels subjected to stability and temper brittleness tests is thus further emphasized.

Merica(11) also states that "precipitation of the hardening constituent is probably always accompanied by some intergranular precipitation. . . In duralumin this leads to intercrystalline corrosion. . . Intergranular precipitation in duralumin is more pronounced during aging at higher temperatures (120 to 200 C., 250 to 390 F.) than at lower or ordinary ones, and in consequence the susceptibility to intercrystalline corrosion and embrittlement may be lessened by employing low aging temperatures, probably a rule of general applicability." It is worthwhile to repeat here that aging of duralumin at room temperature is not thought to be accompanied by precipitation.

Examination of many abnormally failed 4 to 6 per cent chromium tubes failed to reveal any positive evidence of intercrystalline corrosion, which on the other hand can be very easily seen in tubes of 18 per cent chromium, 8 per cent nickel austenitic steel in the same service. This, it is believed, provides another indirect proof that the embrittlement of 4 to 6 per cent chromium tubes is not accompanied by precipitation.

From the evidence presented, it is postulated that the embrittlement of these steels in refinery use or when subjected to aging at about 900 F. (480 C.) occurs without precipitation and is due to structural alteration of the solid solution similar to the aging of duralumin at ordinary temperatures. This may also be the cause of temper brittleness of steels in general. Further possible correlation of this type of aging of 4 to 6 per cent chromium alloys with the temper brittleness of these alloys will be made a subject of further future studies.

CONCLUSIONS

From a practical point of view, the following conclusions can be drawn:

(*a*) The normal failure of 4 to 6 per cent chromium steel in refinery use is usually accompanied by creep, resulting in considerable visible swelling.

(*b*) Abnormal failures result from shock stresses applied to embrittled metal.

(*c*) Embrittlement of 4 to 6 per cent chromium steel can usually be brought about by simple prolonged exposure of most heats of this steel to a temperature of 900 F. (480 C.).

(*d*) Most heats of 4 to 6 per cent chromium show high degree of susceptibility to temper brittleness.

From a theoretical point of view, the following conclusions can be drawn:

(*e*) The embrittlement of 4 to 6 per cent chromium steels is accompanied by no, or at most, a negligible, increase in hardness or tensile strength. Embrittlement is revealed by the drop in impact value.

(*f*) This type of embrittlement in 4 to 6 per cent chromium steels can be brought about by long exposure to temperatures in the neighborhood of 900 F. (480 C.), regardless of the rate of cooling.

(*g*) In some instances, correlation has been found between this type of embrittlement and temper brittleness.

(*h*) The embrittlement of 4 to 6 per cent chromium steel may be due to aging as a result of structural alteration of solid solution, the same as occurs in the aging of duralumin at ordinary temperatures. The evidence that it is not due to a precipitation of some constituent is shown by the improvement in impact value of these steels when broken at 212 F. (100 C.), the absence of hardening, non-aging susceptibility after quenching from near the A_1 point, and, finally, the absence of intercrystalline corrosion in abnormally failed tubes.

(*i*) The possibility is also noted that the temper brittleness of various steels in general may also result from the same type of aging as described in the preceding paragraph.

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DISCUSSION

MR. J. A. MATHEWS.[1]—I wish to compliment Mr. Dixon on his very interesting and instructive paper. He has had considerable experience with 4 to 6 per cent chromium steel. With reference to recovery from brittleness at a temperature of 212 F. (100 C.) I pointed out some years ago a case where the steel improved in impact 1 ft-lb. per deg. Fahr. on heating from room temperature to 150 F. (66 C.). That is remarkable recovery but it is not a cure. Mr. Dixon did not mention that when steel went back to room temperature and was tested cold it was just as brittle as before. To make a permanent recovery, in our experience, it must be reheated to something like 1100 to 1200 F. (595 to 650 C.). I cannot explain that sudden recovery in impact at very low temperatures. I should appreciate some explanation.

MR. E. S. DIXON.[2]—One thing of interest and not readily understood is why a pressure battery with 166 tubes will lose only one or two as a result of knocking when cold rather than lose 50 per cent or more tubes. We are striving to protect our tubes from cold splits. In so doing we are knocking when hot and we heat treat to 1200 F. (650 C.) all tubes in the battery every six months by firing the furnace with no oil flowing through the tubes. Low resistance of impact at room temperatures and those at 212 F. (100 C.) have been mentioned in the last paragraph under the heading Correlation Data of Abnormally Failed Tubes.

The phenomenon of sudden recovery of impact strength at very low temperatures has also puzzled us and, therefore, has been extensively discussed in the text of the paper; we think it must be due to some structural alteration of the metal.

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CREEP PROPERTIES OF 5 PER CENT CHROMIUM, 0.50 PER CENT MOLYBDENUM STEEL STILL TUBES

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Synopsis

Steels of the 5 per cent chromium, 0.50 per cent molybdenum types of two carbon contents have been studied to determine their creep properties. The specimens were cut from finished still tubes to represent both longitudinal and transverse directions. Five heat treatments, representing normalizing and slow cooling from 1550 F. (845 C.) and 2100 F. (1150 C.), were used to produce different structures and grain sizes. Hardness, impact resistance, susceptibility to temper brittleness, and short-time tensile properties at room temperature, 1000, 1100, and 1200 F. (540, 595, and 650 C.) were determined.

Creep properties at 1100 F. (595 C.) for specimens slowly cooled from 1550 F. (845 C.) and the relative creep properties at one stress at this temperature for the four other heat treatments were determined for steels of both carbon contents. A few creep data at 1200 F. (650 C.) are also included. The following conclusions have been drawn:

1. Increase in carbon content materially increased the physical properties and creep resistance.

2. Longitudinal specimens showed better creep resistance than transverse specimens.

3. Better creep properties were obtained for slowly cooled specimens than for normalized specimens.

After the specimens had been subjected to creep tests, the hardness, impact resistance, and short-time tensile properties were determined. From these data it is concluded that subjecting these steels to creep testing at 1100 and 1200 F. (595 and 650 C.) has little effect on the hardness, impact resistance, or tensile properties in short-time tests, thus indicating that these steels are quite stable at 1100 and 1200 F. (595 and 650 C.).

Introduction

When a material like 5 per cent chromium, 0.50 per cent molybdenum steel shows itself fit for a specific and exacting service, like that in oil-still tubes, the user soon requires more than qualitative data collected from service and demands more nearly quantitative data. He recognizes that no laboratory tests can provide design data for this application that will not also require the exercise of engineering judgment, but he welcomes, as more representative, information obtained on material taken from finished still tubes rather than from bar stock of similar composition.

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Among the factors of interest to producer and consumer are the effect of carbon content, of heat treatment and resultant grain size, and of variation of directional properties resulting from working during fabrication.

Desire for information of this type on this steel has been expressed by the Still Tube Subcommittee of the A.S.T.M. - A.S.M.E. Joint Research Committee on the Effect of Temperature on the Properties of Metals.

The Republic Steel Corp. desired much of this information to enable it to produce the most suitable material for this severe service, and in commissioning Battelle Memorial Institute to survey the high-temperature properties, authorized the orienting of the work to cover as completely as feasible matters in which the Still Tube Subcommittee had expressed interest.

While still tube service does not require that deformation in service be held to as low a limit as in many other high-temperature applications, so that a creep rate of say 5 per cent in 10,000 hr. might form a satisfactory basis for comparison of those factors to be studied, and most attention has been paid to such creep rates, it has been attempted also to procure data to show the loads for creep of 1 per cent in 10,000 hr. at 1100 F. (595 C.), for purposes of comparison with other steels.

MATERIALS

The steels used were of the 5 per cent chromium, 0.50 per cent molybdenum type and of two different carbon contents. The analyses of the other alloying elements were duplicated as closely as possible. The two heats were melted in the plant of the Republic Steel Corp., using a 6-ton electric furnace and normal melting practice. The melts were cast in 20-in. round Gathmann type ingot molds. The ingots weighed approximately 4750 lb. each.

The ingots were rolled into blooms and then to 5½-in. rounds. The bars were sent to a tube manufacturer and fabricated into still tubes 5 in. in outside diameter and ⅝ in. in wall thickness. In normal practice the tube manufacturer anneals the still tubes, but in this case the tubes were returned without annealing to permit variations in heat treatment.

Numerous chemical analyses of the two materials gave the following results:

Steel	Chemical Composition, per cent							
	Carbon	Manganese	Sulfur	Phosphorus	Silicon	Nickel	Chromium	Molybdenum
I	0.139	0.38	0.015	0.017	0.35	0.23	4.63	0.54
II	0.181	0.40	0.014	0.015	0.33	0.26	4.96	0.49

Blanks for preparation of longitudinal and transverse test specimens were cut from the finished tubes. To obtain the transverse specimens, short lengths were cut from the still tube, slit lengthwise, heated to about 1700 F. (925 C.) and flattened, admittedly introducing further deformation but not very severe deformation compared to that involved in making the tube.

The major portion of the test data was obtained on the steels given the normal annealing treatment (treatment C). The controlled cooling rate from 1500 to 1300 F. (815 to 705 C.) was for the purpose of duplicating in the small blanks the cooling rate normally attained in cooling a large mass, such as a still tube. The other heat treatments were to evaluate the effects of various annealing temperatures, rates of cooling, grain size, and structure upon the creep and other mechanical properties determined.

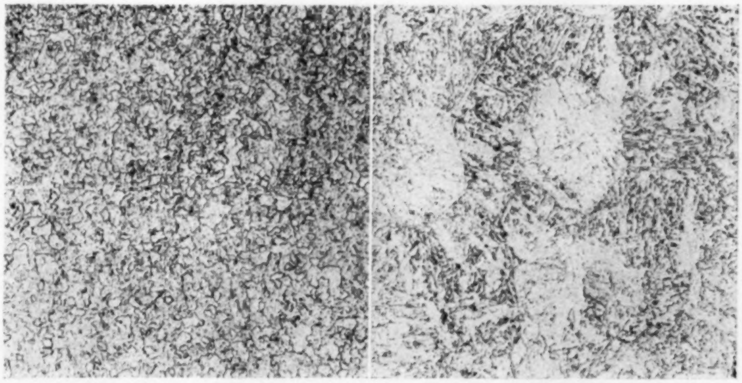
The heat treatments were as follows:

Treatment C......	Normal annealing treatment. 1550 F. (845 C.), one hour, furnace cool to 1500 F. (815 C.), then 25 F. (14 C.) per hr. to 1300 F. (705 C.); then furnace cool to 1000 F. (540 C.); then air cool.
Treatment A......	Same as treatment C, followed by reheating to 1425 F. (775 C.) for 6 hr.; then furnace cool.
Treatment B......	1550 F. (845 C.), one hour, air cool, reheat to 1425 F. (775 C.) for 6 hr.; then furnace cool.
Treatment D......	2100 F. (1150 C.), one hour, air cool, reheat to 1425 F. (775 C.) for 6 hr.; then furnace cool.
Treatment E......	2100 F. (1150 C.), one hour, furnace cool to 1500 F. (815 C.) then 25 F. (14 C.) per hour to 1300 F. (705 C.); then furnace cool to 1000 F. (540 C.); then air cool.

The reheating to 1425 F. (775 C.) was required on the normalized samples to reduce the hardness to comparable values with the slow-cooled samples without changing the grain size resulting from the initial treatment. For comparison this reheating treatment was also added to treatment C. The slow-cooling and normalizing treatments from 2100 F. (1150 C.) were for the purpose of producing maximum structural differences for ease of comparison.

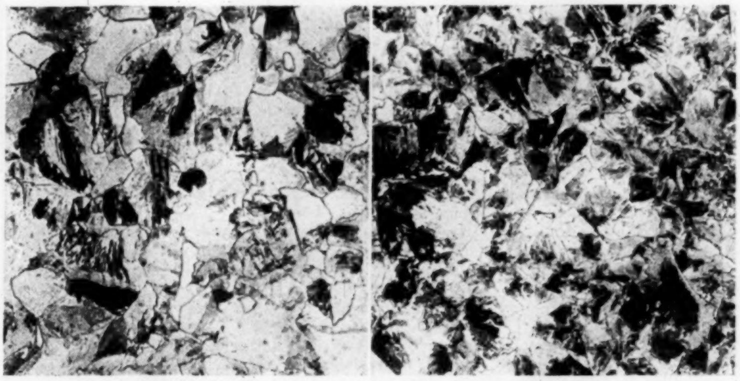
Identification of the various test specimens shows the steel number I or II, the heat treatment A, B, C, D, or E used, the direction of the test specimen in the still tube (longitudinal L or transverse T), and the specimen number, 1, 2, 3, and others. Thus IIC-T4 indicates that specimen is from steel II (higher-carbon heat), given heat treatment C, was a transverse specimen, and was the fourth specimen of its series.

In Fig. 1 are shown photomicrographs of steel IIC (slow cooled) and of steel IID (normalized), whose structures are characteristic of treatments C and A, and B and D, respectively, but totally different from the non-uniform and pearlitic structures shown by steels IE and IIE. Steel IIC clearly shows shading, indicating directional properties, but is otherwise uniform as contrasted with steel IID whose non-uniform structure is due to local softening of hard areas when reheated to 1425 F. (775 C).



Steel IIC
Slow cooled from 1550 F. (845 C.).

Steel IID
Normalized from 2100 F. (1150 C.). Reheated **6 hr. at 1425 F. (775 C.).**

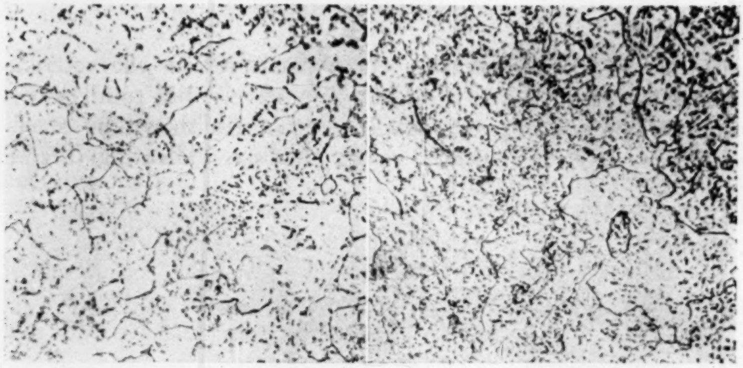


Steel IE
Slow cooled from 2100 F. (1150 C.).

Steel IIE
Slow cooled from 2100 F. (1150 C.).

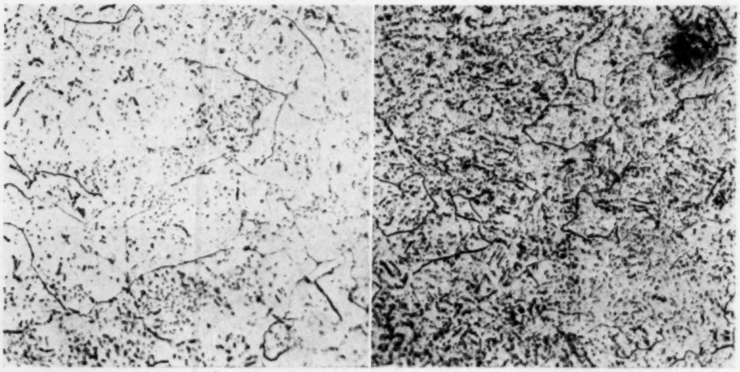
FIG. 1.—Photomicrographs of 5 per cent Chromium, 0.50 per cent Molybdenum Steels Given Various Heat Treatments (× 100).
Etched in 10 per cent Nital.

The photomicrographs at 500 magnifications in Fig. 2 clearly indicate the difference in amount of carbide in steels IC and IIC, but



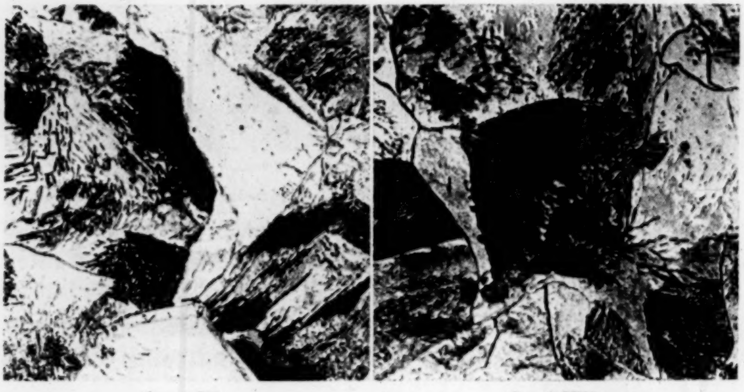
Steel IC Steel IIC

Slow cooled from 1550 F. (845 C.).



Steel IA Steel IIA

Slow cooled from 1550 F. (845 C.). Reheated 6 hr. at 1425 F. (775 C.).



Steel IE Steel IIE

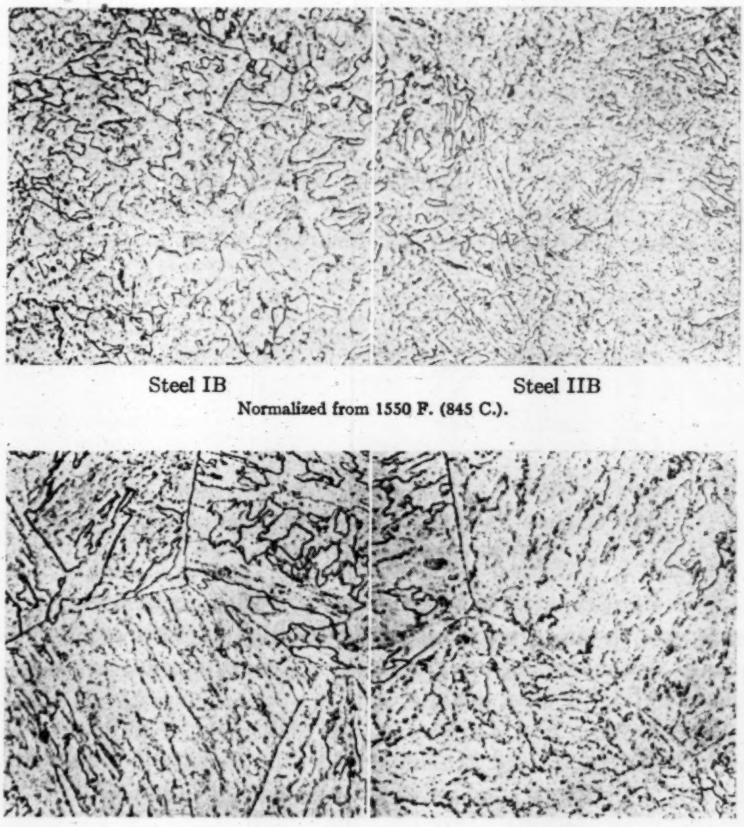
Slow cooled from 2100 F. (1150 C.).

FIG. 2.—Photomicrographs of 5 per cent Chromium, 0.50 per cent Molybdenum Steels Given Various Heat Treatments (× 500).

Etched in 10 per cent Nital.

no discernible difference between steels IA and IC, and IIA and IIC, due to reheating at 1425 F. (775 C.).

The photomicrographs in Fig. 3 show the non-uniformity of structure of steels IB and IIB as contrasted with steels IA and IIA in Fig. 2. The larger grain size of steels ID and IID, as compared with steels IB and IIB, is apparent, and their structures indicate the possibility of structural changes taking place within the grains due to either the normalizing treatment or during the reheating at 1425 F. (775 C.).



Steel IB Steel IIB

Normalized from 1550 F. (845 C.).

Steel ID Steel IID

Normalized from 2100 F. (1150 C.).

FIG. 3.—Photomicrographs of 5 per cent Chromium, 0.50 per cent Molybdenum Steels Given Various Heat Treatments (× 500).

Etched in 10 per cent Nital.

TEST SPECIMENS AND TEST METHODS

Short-time Tests:

A standard 0.505-in. diameter test specimen was used in the room-temperature tension tests. The yield point was determined by the drop of the beam.

A few room-temperature tension tests and all of the short-time tension tests at elevated temperatures, as well as the creep tests, were made on bars with a rectangular test section.

Figure 4 shows details of the test specimen used. This type of test specimen was chosen because it was desired to test a representative section of a tube wall. The tube wall thickness was in the direction of the 0.51 in. dimension; so beginning with a tube of 0.625 in. wall thickness, about 0.055 in. has been machined off each side to obtain the finished test specimen with 0.2 sq. in. section.

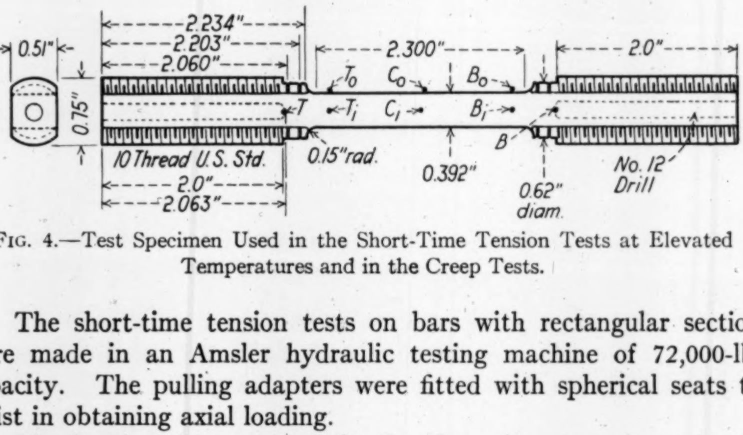


FIG. 4.—Test Specimen Used in the Short-Time Tension Tests at Elevated Temperatures and in the Creep Tests.

The short-time tension tests on bars with rectangular section were made in an Amsler hydraulic testing machine of 72,000-lb. capacity. The pulling adapters were fitted with spherical seats to assist in obtaining axial loading.

The heating furnace used in the short-time tests is 14 in. in length, gap wound, and provided with taps to permit regulation of the current in various sections of the winding to obtain the desired temperature uniformity along the gage length of the test specimen.

A temperature survey of the furnace according to the methods recommended in the Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 – 33T)[1] is shown in Table I. The thermocouples on the outside of the gage length were attached by spot welding.

During the actual tests two thermocouples are used. The thermocouple in the top end beyond the fillet (T) measures the specimen temperature, and the bottom couple beyond the fillet (*B*) is attached to a Wilson-Maeulen automatic temperature controller.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 996 (1933); also 1933 Book of A. S.T.M. Tentative Standards, p. 1030.

Stress-strain curves for determination of the yield strengths were obtained by frames fastened to the adapters and fitted with two Ames dials reading to 0.0001 in. The loading was continuous, and readings of the dials were made simultaneously by two observers. After the yield point was passed the dials were removed and the speed of the head regulated as nearly as possible to 0.25 in. per minute up to the ultimate strength.

The yield strengths were obtained from the stress-strain curves as the stresses at which the materials exhibited 0.2 per cent permanent set according to the Standard Methods of Tension Testing of Metallic Materials (E 8 – 33).[1]

TABLE I.—TEMPERATURE SURVEY OF SHORT-TIME TENSION TEST FURNACE.
POSITION OF THERMOCOUPLES IS INDICATED IN FIG. 4.

Nominal Test Temperature	Top End Beyond Fillet (T)	Top-Gage Length		Center-Gage Length		Bottom-Gage Length		Bottom End Beyond Fillet (B)
		Inside (T_i)	Outside (T_o)	Inside (C_i)	Outside (C_o)	Inside (B_i)	Outside (B_o)	
1000 F. (538 C.)	1000 F. (538 C.)	1000 F. (538 C.)	998 F. (537 C.)	1000 F. (538 C.)	1000 F. (538 C.)	1000 F. (538 C.)	1003 F. (540 C.)	1009 F. (543 C.)
1100 F. (593 C.)	1102 F. (594 C.)	1100 F. (593 C.)	1100 F. (593 C.)	1100 F. (593 C.)	1101 F. (594 C.)	1102 F. (594 C.)	1105 F. (596 C.)	1112 F. (600 C.)
1200 F. (649 C.)	1200 F. (649 C.)	1200 F. (649 C.)	1198 F. (648 C.)	1200 F. (649 C.)	[a]	1202 F. (650 C.)	1203 F. (651 C.)	1209 F. (654 C.)

[a] Couple detached from surface of specimen.

Creep Tests:

The creep-test apparatus used is similar to that used by Kanter and Spring.[2] Details of the creep test equipment and a discussion of procedure and precautions taken for accuracy in creep work at Battelle have been elsewhere discussed.[3,4]

Throughout the series of creep tests herein reported the requirements of the Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 33 T)[5] have been met or exceeded.

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[1] 1933 Book of A.S.T.M. Standards, Part I, p. 949.

[2] J. J. Kanter and L. W. Spring, "'Long-Time' or 'Flow' Tests of Carbon Steels at Various Temperatures, with Particular Reference to Stresses Below the Proportional Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 80 (1928); "Some Long-Time Tension Tests of Steels at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, pp. 110–132 (1930).

[3] H. W. Gillett and H. C. Cross, "Obtaining Reliable Values for Creep of Metals at High Temperatures," *Metals and Alloys*, Vol. 4, July, 1933, pp. 91–98.

[4] H. C. Cross, "High-Temperature Tensile, Creep and Fatigue Properties of Cast and Wrought High and Low Carbon 18 Cr 8 Ni Type Steel from Split Heats," Report of Joint Research Committee of A.S.M.E.-A.S.T.M. on Effect of Temperature on the Properties of Metals, *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, No. 7, July, 1934, p. 533.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 1004 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 1038.

The individual temperature-uniformity calibrations of the various creep test furnaces are not shown, but in every case the variation of temperature over the gage length did not exceed 5 F. (2.6 C.), and in many instances was considerably less.

The telescope micrometer used to measure the elongations is mounted in a slide traveling on a graduated screw and is fitted with a filar micrometer eyepiece. Calibration shows its smallest division to read to 0.0000484 in., which on a specimen with approximately a 2.3-in. gage length gives readings to about 0.00002 in. per inch, or 0.002 per cent.

All Battelle creep apparatus has been modified to permit readings of deformation on opposite sides of the test specimen. The data in this report show the time-deformation curves for the front and back of each test specimen.

Table II.—Ratio of Susceptibility of Temper Brittleness.

Steel	Cooling from Tempering Temperature, 1200 F. (650 C.)	Charpy Impact, ft-lb., Keyhole-Notch Specimen	Ratio of Susceptibility	Izod Impact, ft-lb., V-Notch Specimen	Ratio of Susceptibility
IC-L	Water	57	....	106	....
	Furnace	57	1.0	110	0.96
IC-T	Water	41	....	...	....
	Furnace	45	0.91	...	....
IIC-L	Water	54	....	over 90	....
	Furnace	58	0.93	112	....
IIC-T	Water	37	....	...	....
	Furnace	33	1.1	...	....

After a brief initial period the rates of elongation become practically the same for both sides and the time-deformation curves parallel. After loading and throughout the test period, readings of elongation are made daily by two observers, and the time-deformation curves shown represent averages of the curves of the two observers. Either curve alone follows the average curve too closely to allow reproduction of the separate curves.

Impact Tests:

The impact tests were made on an Amsler combination Izod and Charpy impact-testing machine. It was desired to make impact tests after creep tests. To obtain two impact test specimens from each creep test specimen, the Izod test was chosen. A few Charpy tests were made on material as heat treated.

The impact test specimen had a section 0.394 by 0.380 in., limited to this size by the reduced section of the creep test specimen. The notch was machined in the gage length of the creep specimens about 0.25 in. from the fillet. The end gripped in the anvil of the impact machine had to be shorter than standard, but tests gave comparative results on specimens of short and normal lengths. All Izod tests herein reported, whether on material before or after creep, were made on short-grip specimens. Tests were made in some instances with a V notch and in others with a keyhole notch.

Susceptibility to temper brittleness was checked by means of both Charpy and Izod tests. The susceptibility ratio was found to be practically unity or less in some cases, showing the material to be free of temper brittleness. Duplicate bars were quenched from 1650 F. (900 C.) in water and then reheated for 2 hr. at 1200 F. (650 C.), after which one bar of each steel was water quenched from the tempering temperature and the remainder furnace cooled. The data are shown in Table II.

TABLE III.—PHYSICAL PROPERTIES OF THE TWO STEELS AT ROOM TEMPERATURE AFTER THE VARIOUS HEAT TREATMENTS.

Standard 0.505-in. diameter test specimens.

Steel	Yield Strength,[a] lb. per sq. in.	Tensile Strength, lb. per sq. in.		Reduction of Area, per cent	Elongation in 2 in., per cent	Brinell Hardness Number 3000-kg. load	Izod Impact Resistance, ft-lb.
IC-L	37 700	70 200		72.9	33.7	130	70 to 93
IC-T	32 900	70 700		69.3	33.2	132	....
IIC-L	37 300	77 200		74.5	32.0	138	70 to 78
IIC-T	38 700	77 900		65.1	30.5	142	....
IA-L	49 950	69 400		75.5	31.5	126	90 to 103
IIA-L	55 300	74 200		77.4	32.5	143	100 to 112
IE-L	37 100	70 600		60.1	28.5	136	....
IIE-L	46 580	83 500		53.8	24.5	160	....
IB-L	47 900	76 100		77.0	33.2	152	101 to 109
IIB-L	56 700	82 900		77.9	32.5	167	111 to 117
ID-L	57 300	80 200		76.5	29.7	158	107 to 111
ID-T	57 000	79 600		73.5	32.5	...	....
IID-L	56 700	83 500		71.4	31.0	163	111 to 116
IID-T	57 600	82 900		71.8	33.7	...	....
			Breaking Strength[b]				
IC-L[c]	27 750[d]	67 700	162 000	75.5	39.0	...	....
IC-T[c]	30 500[d]	69 800	165 000	73.1	37.5	...	....
IIC-L[c]	29 350[d]	72 650	180 500	78.7	38.3	...	....
IIC-T[c]	30 600[d]	74 050	165 500	71.8	36.3	....	.. ..

[a] Yield strength determined by drop of beam.

[b] Load at moment of fracture divided by reduced area.

[c] Test specimens with rectangular reduced section as used in short-time tests at elevated temperatures and in creep tests.

[d] Stress to produce permanent set of 0.2 per cent.

TEST DATA

Short-time Tension Tests and Impact Tests:

The tension test data at room temperature on round and rectangular section bars given the various heat treatments are shown in Table III. The values shown are averages for two bars tested.

The specimens given the normalizing treatments B and D show higher tensile strengths, yield strengths, Brinell hardness, and impact resistance than the specimens slow-cooled (treatments C, A and E) from the same temperatures. Only minor differences in elongation and reduction of area are noted for the various heat treatments. Slow cooling from 1550 F. (845 C.) using treatment C produces the lowest properties; the yield strength in particular is much lower than for the other treatments.

Additional 6 hr. heating at 1425 F. (775 C.) followed by slow cooling (treatment A) raises the yield strength and impact resistance with only a slight reduction in tensile strength, with little or no change in hardness and ductility. It will be recalled from the photomicrographs in Fig. 2 that there is no noticeable difference in structures for treatments C and A. Slow cooling from 2100 F. (1150 C.) produces practically the same physical properties in the low-carbon steel I as when slow cooled from 1550 F. (845 C.), but steel II shows a much higher yield strength as determined on round bars by the drop-of-the-beam method.

TABLE IV.—SHORT-TIME TENSION TEST DATA FOR STEELS I AND II. SLOW COOLED FROM 1550 F. (845 C.).

Steel	Temperature of Test		Yield Strength,[a] lb. per sq. in.	Tensile Strength, lb. per sq. in.	Breaking Strength,[b] lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
	deg. Fahr.	deg. Cent.					
IC-L	1000	540	17 100	41 300	97 000	46.0	79.9
IC-T	1000	540	17 600	42 600	93 000	39.0	76.6
IIC-L	1000	540	18 750	44 700	105 000	45.0	83.1
IIC-T	1000	540	19 200	45 600	98 000	38.5	76.5
IC-L	1100	595	15 150	32 500	85 000	45.5	86.5
IC-T	1100	595	16 000	34 000	89 000	48.8	85.8
IIC-L	1100	595	16 900	36 300	98 000	42.3	87.3
IIC-T	1100	595	16 750	35 700	89 000	44.0	85.1
IC-L	1200	650	12 600	25 100	88 000	66.8	87.2
IC-T	1200	650	12 850	26 100	75 000	58.3	89.9
IIC-L	1200	650	13 100	26 600	92 000	55.8	87.9
IIC-T	1200	650	14 000	27 900	81 000	55.8	90.4
IC-L7[c]	1100	595	14 500	31 900	74 000	51.0	85.0
IIC-L6[c]	1100	595	16 000	34 800	86 000	45.0	87.5
IC-L8[d]	1200	650	11 500	23 300	69 000	50.0	89.8
IIC-L4[d]	1200	650	13 000	24 500	79 000	54.0	92.1

[a] Stress to produce permanent set of 0.2 per cent.

[b] Load at moment of fracture divided by reduced area.

[c] After creep test at 1100 F. (595 C.), as indicated in Tables V and VI.

[d] After creep test at 1200 F. (650 C.), as indicated in Tables V and VI.

At the bottom of Table III are shown tensile strength data at room temperature for bars of both steels given treatment C and tested with a rectangular breaking section as used in the short-time tests at elevated temperatures and in the creep tests.

Tensile strengths on the rectangular bars are a little lower, particularly on the higher-carbon steel II. The yield strengths were 4000 to 10,000 lb. per sq. in. lower when determined from the stress-strain curves at 0.2 per cent permanent set than when determined on the round bars by drop of beam. The values for elongation and reduction of area are a little higher for the rectangular bars.

The data for impact resistance of the two steels as heat treated are shown in Tables V and VI. Both steels show Izod impact resistance on a bar slightly smaller than standard of 100 ft-lb. or more for longitudinal material given the A, B, C, and D heat treatments.

TABLE V.—CREEP TEST DATA FOR 0.139 PER CENT CARBON, 4.63 PER CENT CHROMIUM, 0.54 PER CENT MOLYBDENUM STEEL (STEEL I).

Specimen Number as Shown on Curves	Temperature of Test		Load, lb. per sq. in.	Duration of Test, hr.	Initial Elongation, per cent		Secondary Elongation, per cent per hr.	Final Elongation, per cent		Izod Impact Resistance After Creep Test[a], ft-lb.		Rockwell Hardness Number, "B" scale, $\frac{1}{16}$-in. ball, 100-kg. load
	deg. Fahr.	deg. Cent.			Front	Back		Front	Back	Keyhole-Notch Specimen	V-Notch Specimen	
IC-L........	[b]	[b]	......	......	......	......	....	......	......	46	101	73
IC-L7........	1100	595	4 100	1 271	0.027	0.012	0.000105	0.204	0.183	[c]	[c]	....
IC-L6........	1100	595	4 700	1 180	0.072	0.011	0.000182	0.333	0.310	42	....	71
IC-L4........	1100	595	5 250	1 445	0.041	0.026	0.000417	0.753	0.732	44	....	72
IC-L1........	1100	595	7 500	362	0.021	0.055	0.0065	2.733	2.764	44	....	72
IC-L3........	1100	595	10 000	47	0.072	0.076	>0.08	3.932	3.930	43	....	71
IC-T........	[b]	[b]	......	......	......	......	....	......	......	46	71	74
IC-T3........	1100	595	3 250	1 346	0.006	0.025	0.000044	0.124	0.159	....	61	75
IC-T2........	1100	595	5 250	835	0.053	0.015	0.00089	0.835	0.800	43	....	72
IC-T1........	1100	595	6 040	742	0.036	0.036	0.0015	1.273	1.264	44	....	71
ID-L........	[b]	[b]	......	......	......	......	....	......	......	....	104	83
ID-L2........	1100	595	5 250	1 180	0.069	0.004	0.00053	0.867	0.890	46	....	81
ID-T1........	1100	595	5 250	721	0.029	0.031	0.000532	0.619	0.618	...	83	83
IA-L........	[b]	[b]	......	......	......	......	....	......	......	....	101	73
IA-L1........	1100	595	5 250	1 177	0.033	0.031	0.00052	0.729	0.765	....	99	76
IB-L........	[b]	[b]	......	......	......	......	....	......	......	....	111	81
IB-L1........	1100	595	5 250	336	0.033	0.038	0.001930	0.912	0.890	....	110	81
IE-L........	[b]	[b]	......	......	......	......	....	......	......	....	11	76
IE-L2........	1100	595	5 250	1 205	0.07	0.014	0.000030	0.185	0.077	....	12	75
IC-L8........	1200	650	3 000	1 482	0.011	0.022	0.000426	0.680	0.665	[c]	[c]	....
IC-T4........	1200	650	3 000	1 176	0	0.019	0.000976	0.952	0.960	....	50	74

[a] Impact specimens 0.380 by 0.394 in. with notch in direction of 0.394 in. dimension. Depth of notch reduced to 0.065 to give standard breaking section.

[b] Tested for impact resistance as heat treated. Not subjected to creep test.

[c] Used for short-time tension test at same temperature as creep test. For results, see Table IV.

The transverse materials show a lower impact resistance ranging from about 70 to 80 ft-lb. when given the C treatment. Both steels given the E treatment (slow cool from 2100 F., 1150 C.), show very low impact resistance, ranging from 7 to 10 ft-lb. This low value would prohibit the use of material so heat treated.

The tension test data at 1000, 1100 and 1200 F. (540, 595 and 650 C.) for specimens given the C treatment are shown in Table IV.

and the data for both room temperature and elevated temperatures are summarized in Fig. 5. The values shown are averages for two bars tested. Steel II with the higher carbon content usually shows higher yield and tensile strengths than steel I at all temperatures, as does the transverse material when compared with the longitudinal material of each steel.

TABLE VI.—CREEP TEST DATA FOR 0.181 PER CENT CARBON, 4.96 PER CENT CHROMIUM, 0.49 PER CENT MOLYBDENUM STEEL (STEEL II).

Specimen Number as Shown on Curves	Temperature of Test		Load, lb. per sq. in.	Duration of Test, hr.	Initial Elongation, per cent		Secondary Elongation, per cent per hr.	Final Elongation, per cent		Izod Impact Resistance After Creep Test[a], ft-lb.		Rockwell Hardness Number, "B" scale, $\frac{1}{16}$-in. ball, 100-kg. load
	deg. Fahr.	deg. Cent.			Front	Back		Front	Back	Keyhole-Notch Specimen	V-Notch Specimen	
IIC-L......	[b]	[b]	......	......	......	......	....	......	......	46	110	76
IIC-L6......	1100	595	5 100	1 607	0.023	0.025	0.000114	0.316	0.326	[c]	[c]	....
IIC-L3......	1100	595	6 060	1 441	0.028	0.055	0.000268	0.485	0.563	44	....	76
IIC-L2......	1100	595	7 500	362	0.006	0.010	0.0023	1.015	0.887	46	....	77
IIC-L1......	1100	595	10 000	22	......	......	>0.05	1.348	1.239	46	....	77
IIC-T......	[b]	[b]	......	......	......	......	....	......	......	48	76	78
IIC-T1......	1100	595	6 060	1 099	0.048	0.019	0.00041	0.64	0.63	43	....	79
IIC-T2......	1100	595	5 250	1 253	0.025	0.032	0.000228	0.387	0.372	43	....	76
IIC-T4......	1100	595	4 800	1 596	0.031	0.019	0.000068	0.230	0.164	...	54	79
IID-L......	[b]	[b]	......	......	......	......	....	......	......	....	101	86
IID-L2......	1100	595	6 065	1 180	0.042	0.044	0.00042	0.840	0.773	48	....	84
IID-T2......	1100	595	6 065	1 010	0.075	0.015	0.00072	1.103	1.081	....	77–93	81
IIA-L......	[b]	[b]	......	......	......	......	....	......	......	....	106	80
IIA-L1......	1100	595	6 060	1 298	0.025	0.039	0.00025	0.430	0.434	....	107	78
IIB-L......	[b]	[b]	......	......	......	......	....	......	......	....	117	85
IIB-L1......	1100	595	6 060	293	0.050	0.035	0.00218	0.972	1.020	....	115	85
IIE-L......	[b]	[b]	......	......	......	......	....	......	......	....	7	91
IIE-L2......	1100	595	6 060	1 205	0.035	0.035	0.000035	0.145	0.142	....	6	83
IIC-L4......	1200	650	3 000	1 201	0.008	0.017	0.000744	0.624	0.620	[c]	[c]	....
IIC-T3......	1200	650	3 000	1 201	0.021	0.025	0.00085	0.768	0.785	....	52	76

[a] Impact specimens 0.380 by 0.394 in. with notch in direction of 0.394 in. dimension. Depth of notch reduced to 0.065 in. to give standard breaking section.

[b] Tested for impact resistance as heat treated. Not subjected to creep test.

[c] Used for short-time tension test at same temperature as creep test. For results, see Table IV.

Creep Tests:

The time-deformation curves for the creep tests at 1100 and 1200 F. (595 and 650 C.) are shown in Figs. 6, 7, 8, 9, and 10. The details of all the creep tests are shown in Tables V and VI. The majority of the creep tests were made at 1100 F. (595 C.); tests at 1200 F. (650 C.) were made on the steels with treatment C at one load only.

The omission of the points on the time-deformation curves for specimens IIC-L6 and IIC-T4 in Fig. 7 was due to trouble with the elongation measuring equipment. The tests were continued, the

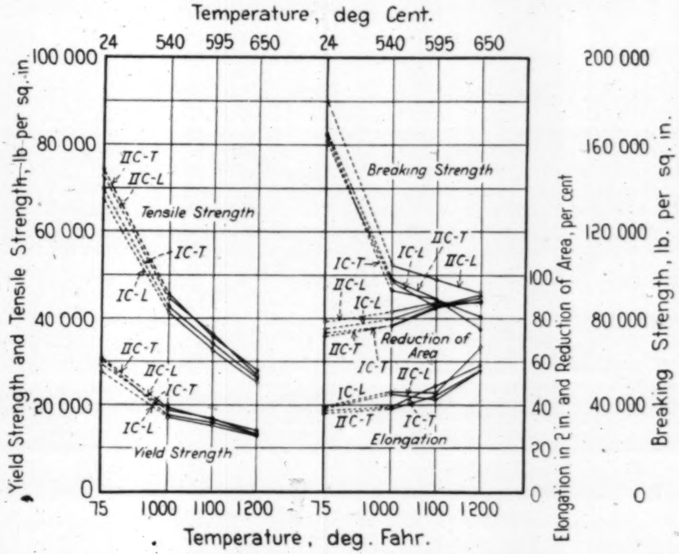

FIG. 5.—Tension Test Data for 5 per cent Chromium, 0.50 per cent Molybdenum Steels at Room Temperature (75 F., 24 C.), 1000, 1100 and 1200 F. (540, 595 and 650 C.).

Longitudinal (L) and transverse (T) specimens. Steel I—0.139 per cent carbon. Steel II— 0.181 per cent carbon. Treatment C—slow cooled from 1550 F. (845 C.).

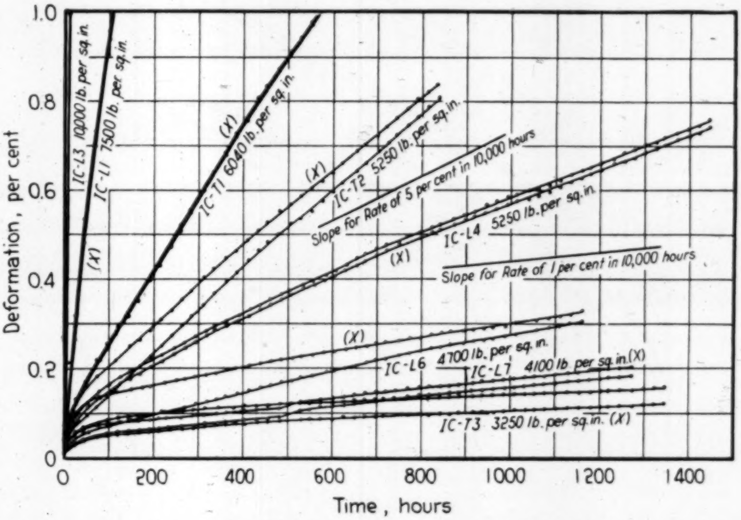

FIG. 6.—Time-Deformation Curves at 1100 F. (595 C.) for 5 per cent Chromium, 0.50 per cent Molybdenum Steel.

Longitudinal (L) and transverse (T) specimens. Steel I—0.139 per cent carbon. Treatment C—slow cooled from 1550 F. (845 C.).

NOTE.—(X) signifies the inside surface of the still tube.

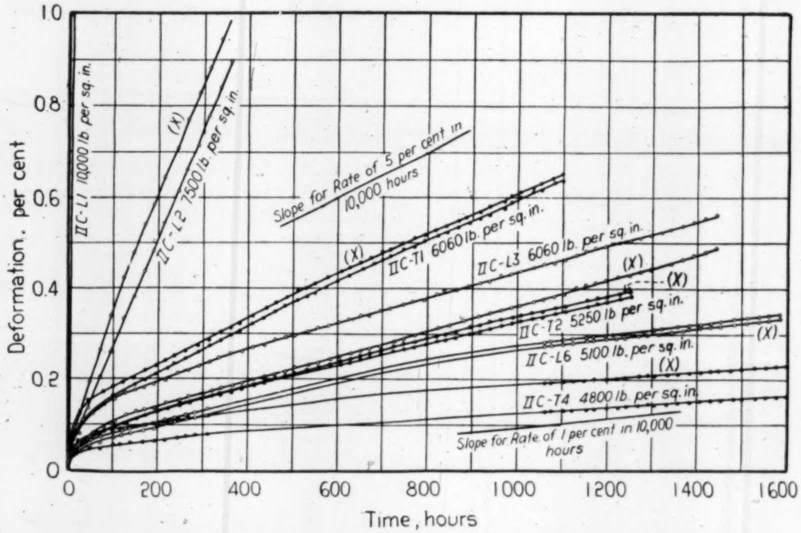

FIG. 7.—Time-Deformation Curves at 1100 F. (595 C.) for 5 per cent Chromium, 0.50 per cent Molybdenum Steel.

Longitudinal (L) and transverse (T) specimens. Steel II—0.181 per cent carbon. Treatment C—slow cooled from 1550 F. (845 C.).

NOTE.—(*X*) signifies the inside surface of the still tube.

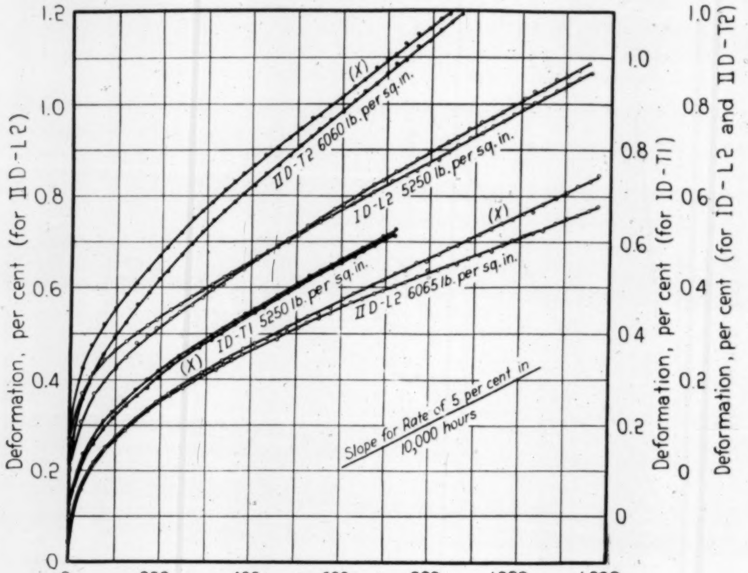

FIG. 8.—Time-Deformation Curves at 1100 F. (595 C.) for 5 per cent Chromium, 0.50 per cent Molybdenum Steels.

Longitudinal (L) and transverse (T) specimens. Steel I—0.139 per cent carbon. Steel II—0.181 per cent carbon. Treatment D—Normalized from 2100 F. (1150 C.), reheated 6 hr. at 1425 F. (775 C.).

NOTE.—(*X*) signifies the inside surface of the still tube.

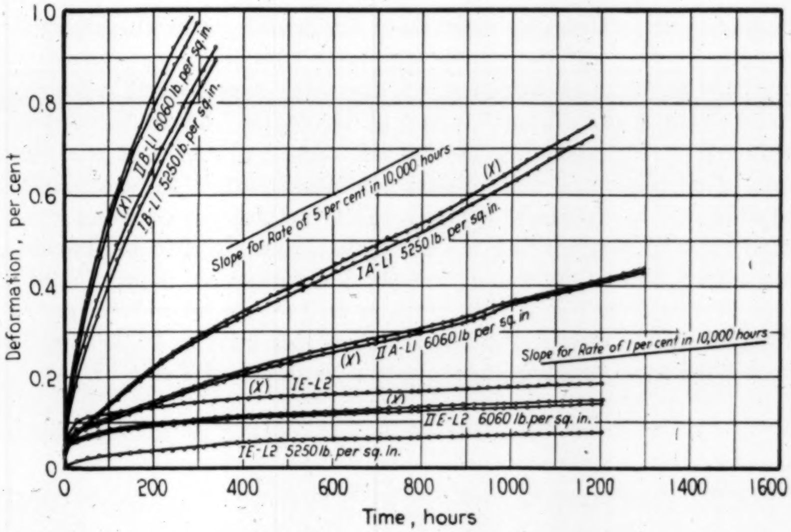

FIG. 9.—Time-Deformation Curves at 1100 F. (595 C.) for 5 per cent Chromium, 0.50 per cent Molybdenum Steels.

Longitudinal (L) specimens. Steel I—0.139 per cent carbon. Steel II—0.181 per cent carbon. Treatment A—slow cooled from 1550 F. (845 C.), reheated 6 hr. at 1425 F. (775 C.). Treatment B—normalized from 1550 F. (845 C.), reheated 6 hr. at 1425 F. (775 C.). Treatment E—slow cooled from 2100 F. (1150 C.).

NOTE.—(*X*) signifies the inside surface of the still tube.

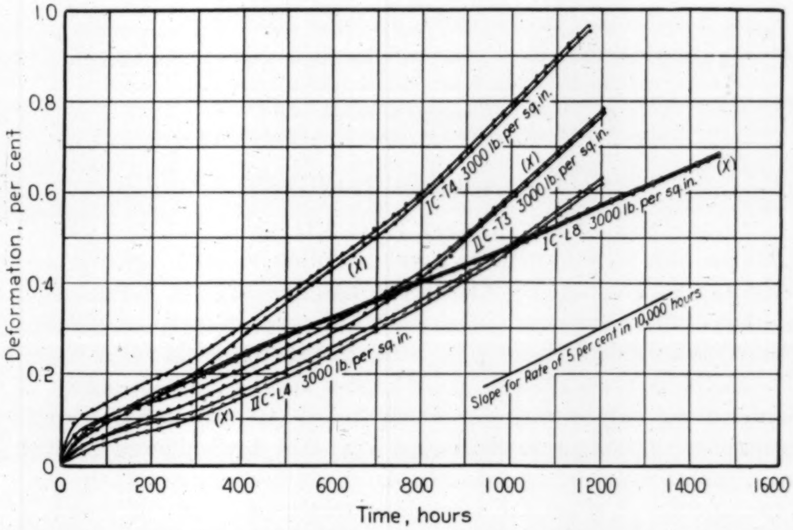

FIG. 10.—Time-Deformation Curves at 1200 F. (650 C.) for 5 per cent Chromium, 0.50 per cent Molybdenum Steels.

Longitudinal (L) and transverse (T) specimens. Steel I—0.139 per cent carbon. Steel II—0.181 per cent carbon. Treatment C—slow cooled from 1550 F. (845 C.).

NOTE.—(*X*) signifies the inside surface of the still tube.

temperatures and loads maintained meanwhile, and the difficulty corrected.

In Fig. 11 are shown data for the total deformations calculated to result from the various stresses in 10,000 hr. These values were obtained by extrapolating the time-deformation curves at the same rate of elongation as existed over the latter part of the test period, as indicated in Tables V and VI. This method gives a lower stress than the stress producing a secondary elongation rate of the same magnitude, since the values for total deformation depend on the magnitude of the initial elongation as well as the secondary rates of elongation. However, the stresses for 1 per cent in 10,000 hr. are nearly the same, using both methods as is shown in Table VII.

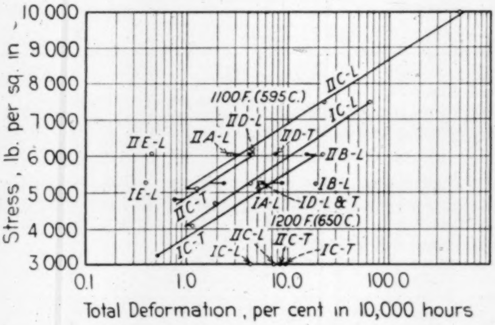

FIG. 11.—Effect of Stress on Total Deformation in 10,000 hr. for 5 per cent Chromium, 0.50 per cent Molybdenum Steels.

For heat treatments, see text, page 3.
Steel I—0.139 per cent carbon. Steel II—0.181 per cent carbon.

DISCUSSION OF TEST DATA

Creep Tests.—Figure 11 and Table VII show that steel II with 0.181 per cent carbon, with only one exception, showed superior creep resistance as compared with steel I with the slightly lower carbon content of 0.139 per cent. There is a difference in creep resistance of about 800 to 1000 lb. per sq. in. between the two steels.

In the tests at 1200 F. (650 C.) at a load of 3000 lb. per sq. in. occurred the only instance in which steel I (treatment C) showed a lower rate of elongation than steel II. The tests showed that the longitudinal specimens possessed better creep resistance than transverse specimens given the same heat treatment. The difference in properties was small, however, and as shown in Table VII the stresses for 1 per cent in 10,000 hr. varied only 300 to 350 lb. per sq. in., or less than 10 per cent.

For comparing the effects of the various heat treatments, creep tests were run at one load only, estimated to produce deformations of 5 per cent in 10,000 hr. For steel I the tests were run at 5250 lb. per sq. in. and for steel II at 6060 lb. per sq. in. Tables V and VI give the rates of elongation, while Table VIII and Fig. 11 show the total deformation estimated to result in 10,000 hr.

The creep resistance of the specimens given the C and A treatments seems about equal. Apparently the reheating at 1425 F. (775 C.) for 6 hr. in treatment A has had little effect on creep properties. This is of particular interest because it indicates that in service short periods of overheating would cause little effect on the creep properties of materials given treatment C. This was expected, since the photomicrographs showed little difference between the two treatments.

TABLE VII.—CREEP DATA FOR STEELS GIVEN ANNEALING TREATMENT C—TESTED AT 1100 F. (595 C.).

Steel	Stress to Produce Secondary Rate of Elongation of 1 per cent in 10,000 hr., lb. per sq. in.	Stress to Produce Total Deformation of 1 per cent in 10,000 hr., lb. per sq. in.
IC-L	4200	4100
IC-T	3850	3800
IIC-L	5170	5100
IIC-T	4850	4850

TABLE VIII.—ESTIMATED PERCENTAGE TOTAL DEFORMATION IN 10,000 HR. AT 1100 F. (595 C.).

	Treatment C, Slow cool—1550 F. (845 C.)	Treatment A, Slow cool—1550 F. (845 C.), reheat 6 hr., 1425 F. (775 C.)	Treatment E, Slow cool—2100 F. (1150 C.), reheat 6 hr., 1425 F. (775 C.)	Treatment B, Normalize—1550 F. (845 C.), reheat 6 hr., 1425 F. (775 C.)	Treatment D, Normalize—2100 F. (1150 C.), reheat 6 hr., 1425 F. (775 C.)
Stress—5250 lb. per sq. sq. in., Steel I	4.31	5.31	0.40	20	5.55
Stress, 6060 lb. per sq. in., Steel II	2.81	2.61	0.45	22	4.51

The specimen given treatment B (normalized from 1550 F., 845 C., reheated 6 hr. 1425 F., 775 C.), showed the poorest creep resistance. This indicates slow cooling rather than fast cooling to be preferred to obtain best creep properties in the 5 per cent chromium, 0.50 per cent molybdenum steel.

The specimen given treatment D (normalized from 2100 F., 1150 C., reheated 6 hr. 1425 F., 775 C.), showed better creep resistance than treatment B as normalized from the lower temperature. However its creep resistance was not equal to that given by the normal anneal (treatment C).

Since the large grain resulting from treatment D gave better creep resistance than treatment B, it was thought advisable to determine the creep resistance of several specimens slow cooled from 2100 F. (1150 C.) with the thought that better creep resistance might be obtained than from treatment C due to the higher heating temperature. This has proved to be the case, for the time-deformation curves shown in Fig. 9 and the data in Table VIII indicate a lower rate of elongation for specimens given treatment E than for any of the other four treatments. However, it should be remembered that treatment E produced a very low impact resistance which would prohibit its use regardless of its superior creep resistance.

These data suggest that possibly better creep resistance than produced by treatment C (slow cool from 1550 F., 845 C.) could be secured by slow cooling from a somewhat higher temperature than 1550 F. (845 C.), which would not at the same time affect the impact resistance of the material appreciably. It should be remembered that the excessively high temperatures were used only to produce maximum structural differences so that good comparisons could be made.

Physical Properties After Creep Test.—Photomicrographs were made after creep test of the structure of specimens IC-T3 (1346 hr. at 1100 F., 595 C., loaded to 3250 lb. per sq. in.) and IIC-T4 (1596 hr. at 1100 F., 595 C., loaded to 4800 lb. per sq. in.). There was no detectable change in the microstructure from that shown in Figs. 1 and 2 for the two steels with treatment C before test. This was as expected, because slow cooling should tend to produce a stable structure. As no differences were noted, the photomicrographs are not shown.

Short-time tension tests were made on four longitudinal specimens at the same temperature at which they were tested for creep. The results are shown at the bottom of Table IV. There were only small reductions in yield and ultimate strengths and ductility values. These data further indicate the stability of the two steels given treatment C.

Izod impact tests using both keyhole and V notches were made on specimens of all treatments after creep test. The data are shown in Tables V and VI. The V notch seemed more selective, in that it indicated greater differences than the keyhole notch. Loss of impact resistance was less than 10 per cent for longitudinal and transverse specimens of both steels with treatment C tested with keyhole notch after creep test at 1100 F. (595 C.). A larger reduction was noted when using the V notch but the material still showed good ductility. For transverse specimens, whose initial impact resistance was lower than for longitudinal specimens, for steel I the drop was from 71 to 61 ft-lb. and for steel II it was from 76 to 54 ft-lb.

For treatment D, steel I dropped from 104 to 83, and steel II from 101 to 85 ft-lb. after creep test at 1100 F. (595 C.). After creep test at 1200 F. (650 C.) both steels show impact resistance at about 50 ft-lb. as compared with original values of 71 to 76 ft-lb. Specimens of both steels given the A or B treatments show practically no change after creep test at 1100 F. (595 C.). Specimens of both steels given treatment E showed practically no change after creep test at 1100 F. (595 C.) from the very low impact resistance indicated as heat treated.

Rockwell hardness determined before and after creep test on the two steels given the various heat treatments showed little or no change. The values are indicated in Tables V and VI.

Comparison with Published Data.—H. D. Newell[1] has published data obtained by Norton showing 4800 lb. per sq. in. for deformation of 1 per cent in 10,000 hr. for an annealed 4 to 6 per cent chromium, 0.50 per cent molybdenum, 0.20 per cent (maximum) carbon steel tested at 1100 F. (595 C.) The creep data herein reported for the higher carbon steel II show 4850 and 5100 lb. per sq. in. for transverse and longitudinal material respectively, indicating a good check between the two sets of data. The short-time tensile strength data at 1000, 1100 and 1200 F. (540, 595 and 650 C.) shown in Table IV and Fig. 5 check quite closely the data published by Newell,[1] except for percentage elongation, in which case Newell's data are a little higher. The short-time tensile strength data for an annealed and drawn 0.21 per cent carbon, 4.5 per cent chromium, 0.6 per cent molybdenum steel as given by F. B. Foley[2] show a much higher tensile strength and lower elongation. Full details of the heat treatments are not given in either case.

SUMMARY

Increase in carbon content from 0.139 to 0.181 per cent in a 5 per cent chromium, 0.50 per cent molybdenum steel increased the physical properties and creep resistance materially.

Longitudinal material showed better creep resistance than transverse material of either carbon content, but the differences were small and less than 10 per cent.

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[1] H. D. Newell, "Alloy Steel Tubes for Refinery Service," *Refiner and Natural Gasoline Manufacturer*, Vol. 12, No. 4, April, 1933, p. 122.

[2] F. B. Foley, "Characteristics of Steel at Elevated Temperatures," *Refiner and Natural Gasoline Manufacturer*, Vol. 12, No. 5, May, 1933, p. 180.

Better creep properties were obtained from slow-cooled materials as compared with normalized materials.

Uniform structures with well-defined grain were obtained by slow cooling, but normalized and reheated materials of comparable hardness showed less uniform structures.

Impact resistance of transverse material was lower than for longitudinal material.

Impact tests made on specimens subjected to load and temperature for 1000 hr. or more revealed no notable drop in toughness. With some of the structures resulting from initial heat treatment, slight reduction was noted; but even in these cases a large measure of toughness remained.

Acknowledgments.—The advice and helpful suggestions of H. W. Gillett of Battelle Memorial Institute, E. C. Smith and M. J. R. Morris of Republic Steel Corporation are gratefully acknowledged. The authors also wish to thank F. B. Dahle of Battelle for valuable cooperation in the creep-test program and G. Rowland of the Republic Steel Corporation for preparation of samples for microscopic examination.

DISCUSSION

Mr. J. J. Kanter[1] (*by letter*).—The short-time and long-time tension test data which Cross and Johnson have so excellently presented substantiate some results obtained at Crane Co. Laboratory upon a piece of still tubing of the same type of analysis as the two reported upon. Inasmuch as Battelle Memorial Institute and Crane Co. are employing similar apparatus in high-temperature testing, the results are offered for what they are worth in making a comparison of data.

Tests were made upon specimens taken from a section of tubing furnished by the National Tube Co. of 4-in. outside diameter and $\frac{3}{8}$-in. nominal wall thickness, having the following analysis:

Carbon, per cent	0.075	Silicon, per cent	0.05
Manganese, per cent	0.43	Nickel, per cent	0.19
Sulfur, per cent	0.010	Chromium, per cent	5.18
Phosphorus, per cent	0.011	Molybdenum, per cent	0.44

This tubing was tested in the annealed condition as supplied, the hardness being Rockwell, "B" scale, 68.5 (Brinell 122).

Threaded test bars were turned from longitudinally cut strips of tubing. The breaking sections were 0.380 in. in diameter and of 2-in. gage length. The wall thickness of the tubing was somewhat greater than the nominal $\frac{3}{8}$ in. (0.375), which permitted the 0.380-in. diameter.

Short-time tensile properties of this tubing at various temperatures are given below:

Temperature of Test		Yield Strength, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
deg. Fahr.	deg. Cent.				
70	21	36 600 37 000	64 200 64 100	36.5 35.0	70.5 69.8
550	290	28 800	48 900	30.5	70.7
800	425	25 600	43 900	24.0	70.0
1000	540	16 600	37 500	32.0	72.0
1200	650	......	22 800	41.0	79.5

It will be noted that the strength of this tubing is consistently lower at all temperatures than those reported by Cross and Johnson. This is no doubt attributable to its much lower carbon content, 0.075 per cent, as against 0.139 per cent and 0.181 per cent in the materials used by the authors.

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[1] Testing Engineer, Crane Co., Chicago, Ill.

The creep tests for this low-carbon 5 per cent chromium, 0.5 per cent molybdenum tubing were made at three stresses: 30,000; 10,000; and 3000 lb. per sq. in.

The data are represented graphically in the accompanying Fig. 1 showing a comparison of the temperatures at which a total deformation of 1 per cent in 10,000 hr. occurs determined by "step-temperature" procedure with the short-time tensile and yield strength curves. This comparison indicates that at temperatures above 760 F. (405 C.) yield strength is in excess of the 1 per cent in 10,000 hr. creep stress criterion.

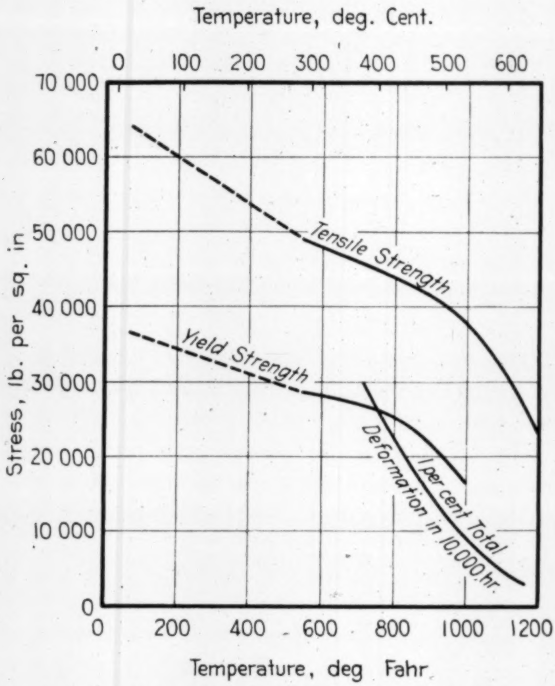

FIG. 1.—Chromium Molybdenum Steel Tubing.

Interpolating upon the creep curve a value of about 4800 lb. per sq. in. is found for 1100 F. (595 C.), the comparison temperature used by the authors. Similar values given by them are 4100 and 5100 lb. per sq. in., respectively, for their two steels. As comparisons of creep data from different sources go, this is fairly good agreement, our value lying within the range of the authors' two values. Summarizing these values with Newell's, and another estimated from determinations by Clark, Taylor and White at 1000 and 1200 F. (540 and 650 C.), we have:

SOURCE	CARBON, PER CENT	STRESS FOR 1 PER CENT TOTAL DEFORMATION IN 10,000 HR. AT 1100 F., LB. PER SQ. IN.
Crane Co	0.075	4800
Cross and Johnson	0.139	4100
Clark, Taylor and White	0.15	4500
Cross and Johnson	0.181	5100
Newell	0.20 (max.)	4800

This comparison indicates a lack of correlation between creep strength and carbon content. A conclusion to the effect that carbon content over the range 0.075 per cent to 0.20 per cent is not a factor in the creep strength, is not warranted. The other variables encountered in creep testing, such as temperature measurement and control, non-uniformity of material, and other differences in methods of test and evaluation of data, may be responsible for discrepancies great enough to make fine-haired analyses of creep data unwarranted. It is encouraging, however, that the four sources of values do not show the great disparity often found between different creep investigators.

MR. F. B. FOLEY[1] (*by letter*).—The authors cite agreement between Norton's 4800 lb. per sq. in. producing 1 per cent in 10,000 hr. creep in a 0.20 per cent carbon steel with their 4850 and 5100 lb. per sq. in. for 0.18 per cent carbon steel. Their 0.18 per cent carbon steel showed about 1000 lb. per sq. in. higher load, for the same deformation, than their 0.14 per cent carbon steel. It seems to me that if an increase of four points in carbon causes 1000-lb. increase in strength, two points might be expected to do something, although even so the comparison is good for creep-testing data. But referring to the comparison of Cross and Johnson's results with those of mine that have been cited, I believe composition and possibly also treatment may explain the difference. The following is the complete data on the steel we tested:

Composition, per cent:

	Carbon	Manganese	Silicon	Chromium	Molybdenum
Foley	0.21	0.65	0.52	4.56	0.57
Cross and Johnson	0.18	0.40	0.33	4.96	0.49

Treatment:

Foley..................1700 F. (925 C.), 1 hr., air cool; 1600 F. (870 C.), 1 hr., slow cool; 1200 F. (650 C.), 1 hr. air cool. Brinell 241.

Cross and Johnson......1550 F. (845 C.), 1 hr., furnace cool to 1500 F. (815 C.), then 25 F. (14 C.) per hr. to 1300 F. (705 C.); then furnace cool to 1000 F. (540 C.); then air cool.

Results:

	Temperature		Tensile Strength, lb. per sq. in.	Proportional Limit, lb. per sq. in.	Yield Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent
	deg. Fahr.	deg. Cent.						
Foley	1100	540	71 900	17 500	......	40 000	29.5	82.2
	1150	565	49 500	10 500	......	24 000	35.5	87.9
Cross and Johnson	1000	540	44 700	......	18 750	......	45.0	83.1
	1100	595	36 300	......	16 900	......	42.3	87.3

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[1] The Midvale Co., Philadelphia, Pa.

At room temperature the authors found that their rectangular tension test specimens gave lower tensile and yield strength and higher ductility than standard 0.505-in. test bars. This also may help to account for the difference in properties, mine having been obtained with standard 0.505-in. test bars.

Mr. C. M. Loeb, Jr.[1]—I should like to ask Mr. Cross how he accounts for the very low impact strength obtained from slow cooling at 2100 F. (1150 C.).

Mr. H. C. Cross.[2]—The only thing we can say is that it is a very coarse pearlitic structure.

Mr. Loeb.—I should not expect such very low impact even from the larger grains with the low Brinell hardness.

Mr. Cross.—We did not expect it, either; we were very much surprised to find it. It is to be regretted that material with such good creep resistance should have such low impact resistance. This suggests a heat treatment such as slow cooling from a higher temperature between 1550 and 2100 F. (845 and 1150 C.), admittedly giving a lower impact resistance than for material slow cooled from 1550 F. (845 C.) but at the same time possibly obtaining better creep properties.

Mr. R. P. DeVries.[3]—I should like to ask whether any tests were made to see whether normal heat treatment, say about 1700 F. (925 C.) oil quench with rather high draw in the neighborhood of 1200 to 1400 F. (650 to 760 C.) would produce any appreciable change in the strength shown in short-time tension tests. Various materials which the Germans recommend for these same purposes are also recommended to be heat treated along the above-mentioned lines.

Mr. Cross.—The short-time tension tests showed higher yield strengths and ultimate strengths without any marked change in ductility for materials that were normalized and drawn as compared with slow-cooled materials. I suppose that equal or better short-time tensile properties would be obtained from materials oil quenched and drawn as were obtained from materials normalized and drawn.

Mr. J. A. Mathews.[4]—In reference to Mr. DeVries' inquiry concerning oil-tempered and drawn steel *versus* steel in the annealed condition, in the oil-still industry, annealed tubes are generally used, whereas for bolts and studs oil-hardened and tempered steel is the common practice. We have found higher creep resistance in the oil-hardened and tempered material for studs, by 1000 or 1500 lb. per sq. in., compared with similar steel to that used in still tubes. I believe the oil-hardened and tempered steel would give better figures than either normalized, or normalized and annealed.

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[2] Metallurgist, Battelle Memorial Institute, Columbus, Ohio.

[3] Director of Research, Ludlum Steel Co., Watervliet, N. Y.

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THE INTERPRETATION OF CREEP TESTS

BY P. G. MCVETTY[1]

SYNOPSIS

Creep tests are so widely used to evaluate metals and alloys for elevated temperature service that it becomes desirable to review the whole subject of their requirements and limitations. In the past ten years, design has advanced rapidly toward higher temperatures, and the materials being offered to meet these new conditions are vastly different from the simple carbon steels formerly used. If it were possible to conduct creep tests under exact simulation of working conditions and over a period of time comparable with the expected service life, there would be little difficulty in interpreting the results. In addition to the prohibitive cost of such a procedure, experience has shown that the materials would be obsolete long before the completion of such a testing program. It is less than twelve years since the results of some of the earliest creep tests were published by Dickenson, while a service life of 20 years is frequently demanded. As a result, creep tests must be extrapolated if they are to have design value and the time ratio may be as great as 100 to 1. Granting all the objections to extrapolation, it may be looked upon as a necessary evil.

This paper discusses the fundamental nature of the creep curve and the probable effect upon it of strain-hardening, annealing, age-hardening, and other phenomena associated with the long exposure of complex alloys to stress at elevated temperatures. The possible effects of "creep-recovery" are mentioned. Examples are given to show how, under suitable conditions, published creep data may be correlated. An attempt is made to stimulate interest in the general subject of interpretation of creep data in the hope that a more intensive study will develop better methods than those commonly used.

INTRODUCTION

The term "creep rate" has become so familiar in the past few years that there is a tendency to ignore the assumptions back of it. The doubts as to its significance which resulted from differences in testing technique have been partially dispelled by the adoption of a test code[2] which, theoretically at least, makes all creep tests comparable. We find, as a consequence, the creep rate taking a similar place in the evaluation of materials at elevated temperatures that the yield strength holds at normal temperature. The designer is eager to accept the term because it can be so readily adapted to his needs. It allows a convenient comparison of materials, a direct basis for the assignment of working stresses and, by suitable mathematical treatment, an indirect solution of many design problems involved in the use of metals and alloys at high temperatures.

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[2] Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 1004 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 1038.

It is a peculiar fact, however, that those who are most familiar with high-temperature testing and with the vagaries of complex alloys under stress at elevated temperatures are somewhat reticent about their interpretation of creep tests. They foresee the possible effects of slight changes in the many variables involved during the tests and in subsequent service, and they hesitate to predict the performance of a new alloy, especially if the allowable deformation must be held within close limits.

Since it is one of the main functions of the Society to bring together the viewpoints of the producer and the consumer, it seems fitting at this time to discuss the interpretation of creep tests. We have on one hand a demand for reliable data which can be used to support the progress of design toward higher temperatures; on the other hand, we find available materials exhibiting strange peculiarities which make it difficult to evaluate them by any simple method. The designer specifies an estimated time-temperature cycle, desired service life and limiting deformation, and asks what stress he may use safely. It is this apparently simple question which has stimulated creep testing for over ten years. The answer is complete only if it gives the allowable stress and cost per pound of all available materials, since the economic side of the problem also must be considered. It is obvious that any change in the specified variables must have its effect upon the allowable stress. To satisfy the need for design information it is necessary, therefore, to establish for each material the relations among the four variables—stress, strain, time, and temperature covering the desired range for each variable.

SIGNIFICANCE AND LIMITATIONS OF THE CREEP RATE

Of the various methods of interpretation that have been proposed, the simplest combines strain and time into creep rate. For a single temperature the logarithm of this rate is then plotted against the stress or the logarithm of the stress giving an approximately straight line and an implied relation expressed by an exponential or power function. These curves permit easy interpolation between test stresses and extrapolation to lower stresses and they have been widely used because of their convenience. The fact that some authorities use a power function while others use an exponential function to express the relation between stress and creep rate at constant temperature shows a lack of data to decide which is correct. Either method has a decided disadvantage in that logarithmic plotting prevents use of the assumption that creep rate is zero when stress is zero. The inference, also, that a definite creep rate may be associated with each combination of stress and temperature is open to criticism. Furthermore, the use of a creep rate in this way suggests no limitation upon the variable, time. In other words, it is assumed that the creep curve is a straight line through the origin and that it may be prolonged indefinitely. It is difficult to reconcile these assumptions with actual creep data. Since our present methods of analysis lead to doubtful conclusions, it is desirable to make a new survey of the problem in the light of available data.

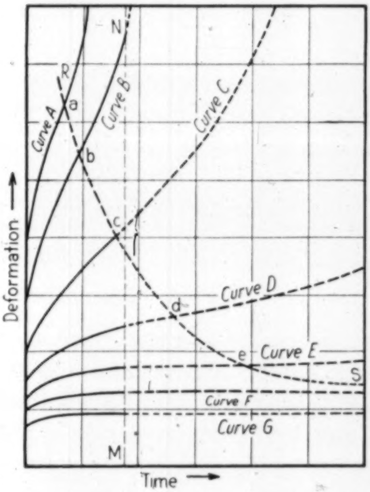

Fig. 1.—Creep Curves for Several Stresses at Constant Temperature.

Interpolation and Extrapolation of Creep Curves

In Fig. 1, curve *A* represents a typical creep curve as obtained at constant temperature and a relatively high stress. An elastic deformation results from the application of the load after which creep proceeds at a gradually diminishing rate. At *a* an inflection occurs after which the creep rate increases until the test specimen breaks. Curve *B* is similar for a lower stress, the longer time to produce fracture resulting from lower creep rates. As the stress is further reduced, similar tests on different specimens give curves

C, D, E, F and *G*. The locus of points of inflection, *RS*, is not well known because tests are seldom carried beyond the line *MN*. Available data indicate, however, that the change from a decreasing to an increasing creep rate occurs at a gradually diminishing value of total deformation represented by the points, *a*, *b*, *c*, *d* and *e*. To determine the inflection points for curves *F* and *G* would require tests of extremely long duration. As the stress decreases, the creep curve is essentially a straight line for a progressively longer period of time as it approaches its inflection point. Theoretically, the rate is continuously decreasing but actually, the line may be considered straight without serious error. The nature of the curves is such that we may expect the increase of rate to be equally slow after the inflection has been passed. Since actual working stresses are usually below curve *G*, the assumption that the creep curve approaches a straight line is sufficiently accurate for many purposes. In no case, however, does the straight line pass through the origin. In other words, its equation must have the form $\epsilon = \epsilon_o + v_o t$ rather than $\epsilon = v_o t$. Any analysis of families of creep curves must make some allowance for the more rapid creep rate in the initial stage.

CREEP OF PURE METALS—STRAIN HARDENING AND ANNEALING

To simplify the discussion of creep phenomena, let us consider that the curves of Fig. 1 represent tests of a pure metal, thus eliminating any possibility of solid solution or precipitation of constituents. For such a material, the decreasing creep rate in the first stage may be attributed to strain hardening which results from the gradually increasing deformation. If it were not for the softening effect of temperature upon strain-hardened material, this might continue indefinitely as the creep rate approached zero.

Studies of the effect of temperature upon strain-hardened material show that the time required to produce softening at a given temperature decreases as the amount of deformation increases. This softening tends to increase the creep rate.

In this ideal case, we have two opposing forces, one decreasing and the other increasing in magnitude as time goes on. It should be noted that separate studies of both the strain-hardening and the annealing effects reveal no discontinuities and a creep curve representing the net effect of the two phenomena must have a point of inflection as shown in Fig. 1. In the absence of a more complete explanation, we may say that the form of the typical creep curve is greatly influenced if not entirely controlled by strain-hardening and annealing during the progress of the test. If practical considerations restrict our tests to that portion of the curves lying to the left of line MN, we may use the reasoning outlined above to supply the dotted portions of the curves representing the longer times found in service. Without some simplification of this kind it would be practically impossible to analyze the very complicated conditions actually existing and make allowance for necessary departures from the assumed simple relations. It must not be inferred, however, that these departures are negligible.

Creep of Complex Alloys—Structural Changes

Recent tests of complex alloys have revealed many peculiarities. For example, impact tests before and after creep tests frequently show marked embrittlement of the material due to long exposure to stress at high temperature. This, in itself, may be enough to prevent the use of the material where good ductility is considered essential. For the purpose of this discussion, it is more important to consider the fact that the data obtained from the creep tests must have been influenced by the gradual structural change within the material. The propriety of extrapolating such a test to cover a long service life is extremely questionable.

Another example recently came to the attention of the author in which a test showed continuous negative creep over a period of 2500 hr. This can only be explained by a structural change causing a decrease of volume which completely masked the natural creep in the direction of the applied stress. One advantage of a test of this kind lies in its warning to investigate structural stability. A more dangerous case is the one in which structural change decreases all the creep rates in a series of tests without showing negative creep. Special precautions are necessary at all times to avoid this possibility if the data are to be used for design purposes.

From these examples, it is apparent that creep tests are vitally affected by structural changes during the progress of the test and their interpretation is affected also by subsequent changes in service. The lack of structural stability of non-ferrous materials has received considerable attention.[1,2] Recent investigations indicate that the alloys recommended for high-temperature applications are subject to similar changes. This introduces a metallurgical problem beyond the scope of the present paper but it is convenient here to divide such changes into three broad classes. In one, the hardness increases gradually to a maximum, and continued exposure to stress and tem-

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[1] P. D. Merica, "The Age Hardening of Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Division, Vol. 99, p. 13 (1932).

[2] F. R. Hensel and E. I. Larsen, "Age-Hardening Copper-Titanium Alloys," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Division, Vol. 99, p. 55 (1932).

perature produces no appreciable further change. The effect of this type of change is similar to that of strain-hardening. If the condition of approximate stability finally reached is not accompanied by embrittlement or other undesirable effects, the creep curve will merely show lower creep rates in the early stage and a longer time to reach the inflection point. This offers no difficulty of interpretation.

The second works in the opposite direction, a familiar example being the gradual spheroidization of cementite which has been studied by Bailey and Roberts[1] and others. If a long life is desired and the allowable deformation is small, the stable condition may be obtained prior to the test by suitable heat treatment, so as to make the extrapolation of the creep curves more reliable. It is evident, however, that it may be possible to secure a long service life before the spheroidization affects the strength seriously. This is equivalent to an increase in the annealing effect mentioned for a pure metal. Careful study of an individual case should allow the use of a higher stress than would be permitted in following hard and fast rules.

The third type of age-hardening involves a gradual increase to a maximum followed by a similar decrease to a condition of approximate stability. Alloys showing this phenomenon during the creep test give results particularly difficult to interpret. This does not prevent their use, however, because it is usually possible to apply a stabilizing treatment prior to the creep tests to bring them into approximate equilibrium with test and service conditions. Any attempt to take advantage of a temporary improvement in creep properties due to this type of age-hardening should be avoided, if allowable deformation is the criterion of safety.

From this it is evident that the interpretation of creep tests requires some familiarity with the characteristics of the materials and such auxiliary tests or preliminary heat treatment, or both, as may be found necessary to justify extrapolation. If a family of creep curves is interpreted without considering the possibility of lack of stability, the results may be decidedly misleading. If a published creep rate for a given stress and temperature is accepted without question and used for design purposes the danger of improper interpretation is further increased.

It has been suggested[2] that the entire responsibility of interpretation be placed upon the designer. This imposes a serious additional burden upon the factor of safety and it may lead to the elimination of desirable alloys as a result of improper interpretation and application. It would seem fairer to all concerned if the evaluation of materials resulted from the closest cooperation between the metallurgist and testing engineer. It is obvious that best results can be obtained by bringing together full knowledge of service conditions, metallurgical characteristics of available alloys and the results of suitably chosen tests. This cannot be accomplished by a mere tabulation of creep rates for design use.

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[1] R. W. Bailey and A. M. Roberts, "Testing of Materials for Service in High Temperature Steam Plant," *Proceedings*, Inst. Mechanical Engrs., Vol. 122, p. 209 (1932).

[2] H. W. Gillett and H. C. Cross, "Obtaining Reliable Values for Creep of Metals at High Temperatures," *Metals and Alloys*, Vol. 4, No. 7, July, 1933, p. 91.

It is out of the question to suggest that further progress in design wait for the better test data which may be expected from the use of the new test code.[1] In the meantime, the maximum use must be made of the data from various sources now available. These data have been acquired at considerable expense in various laboratories in this country and abroad. In many cases, fairly reliable interpretation is possible; in others it is inevitable that misleading conclusions may be reached. By suitable correlation of data, tests notably out of line may be weeded out and replaced by a few carefully chosen check tests. In this survey nothing can take the place of sound engineering judgment.

TABLE I.—CREEP DATA ON CAST AND FORGED STEEL (TAPSELL AND JOHNSON).

Stress, lb. per sq. in.	Final Strain, ϵ, in. per inch	Time, t, hr.	Minimum Creep Rate, v_0, in. per inch per hr.	v_0t, in. per inch	Initial Elastic Plus Plastic Strain, $\epsilon_0 = \epsilon - v_0t$, in. per inch	Elastic Strain, ϵ', in. per inch	Initial Plastic Strain, ϵ_0'', in. per inch
0.30 PER CENT CARBON CAST STEEL AT 400 C. (750 F.).							
35 840	0.0368	960	0.0000125	0.01200	0.0248	0.0014	0.0234
33 600	0.0267	720	0.0000072	0.005184	0.0215	0.0013	0.0202
31 360	0.0209	864	0.0000050	0.004320	0.0166	0.0012	0.0154
29 120	0.0163	1368	0.0000030	0.004104	0.0122	0.0011	0.0111
25 760	0.0072	912	0.00000148	0.001350	0.00585	0.0010	0.00485
22 400	0.0036	1656	0.000000375	0.000621	0.00298	0.00087	0.00211
0.39 PER CENT CARBON FORGED STEEL AT 400 C. (750 F.).							
42 560	0.0229	504	0.00001230	0.0061992	0.0167	0.00174	0.01496
35 840	0.0104	1080	0.00000268	0.0028944	0.0075	0.00146	0.00604
29 120	0.0055	4128	0.000000375	0.0015480	0.0040	0.00119	0.00281

It has been stated that the designer requires a knowledge of the relations among the four variables—stress, strain, time and temperature. The first two are covered in the conventional short-time tension test,[2] the first three in the creep test[1] and the fourth by conducting short-time tension and creep tests at suitably chosen temperatures.

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[1] Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 1004 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 1038.

[2] Tentative Method of Test for Short-Time High-Temperature Tension Tests of Metallic Materials (E 21 - 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 996 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 1030.

Creep Recovery—Effect of Stress Changes

A paper on this subject would be incomplete without reference to the possibility of "creep recovery." It has been known for several years that removal of the stress in the creep test may be followed by a recovery of part of the creep. A recent paper[1] shows a creep test of a nickel-chromium-molybdenum steel at 450 C. (840 F.) and a stress of 22,400 lb. per sq. in. Reduction of the stress to 896 lb. per sq. in. for a period of 500 hr. caused a recovery of about 60 per cent

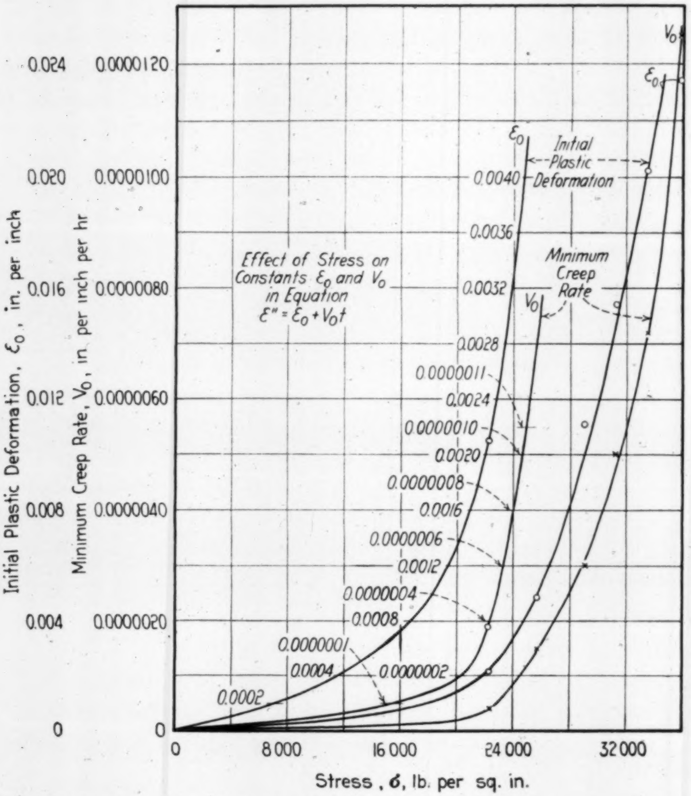

Fig. 2.—National Physical Laboratory Creep Tests of 0.30 per cent Carbon Cast Steel.

Annealed 800 C. (1470 F.), 19 hr. Rough machined. Annealed 550 C. (1020 F.), 6 hr. Tested at 400 C. (750 F.) (H. J. Tapsell and A. E. Johnson, *Special Report No. 17*, Department of Scientific and Industrial Research (1931).

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[1] H. J. Tapsell and L. E. Prosser, "High Sensitivity Creep Testing Equipment at the National Physical Laboratory," *Engineering*, February 23, 1934, p. 212.

of the creep which had occurred in the first 1500 hr. The author found similar results in tests of a medium-carbon steel in 1925. This is extremely important for applications in which the stress is not continuously applied because a single long period at reduced stress would not be expected to have the same effect upon the creep curve as several shorter periods involving the same total time. The interpretation depends also upon the material because any structural instability affected by exposure to stress and high temperature would also be affected by a change in the stress. Under proper control, creep recovery offers an opportunity to increase the working stress in intermittent stress applications, but it is obvious that this advantage must be used advisedly. In a recent paper,[1] the author has suggested a method of attack upon the miscellaneous creep data now available. The main purpose of such a survey will be to reduce all data to a comparable form to allow correlation.

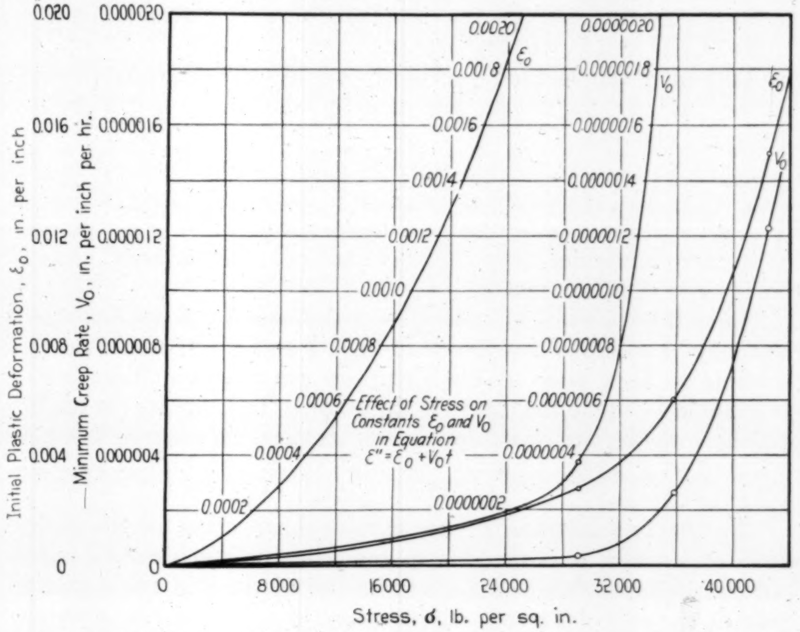

FIG. 3.—National Physical Laboratory Creep Tests of 0.39 per cent Carbon Forged Steel.

Furnace cooled. Rough machined. Annealed 550 C. (1020 F.). Tested at 400 C. (750 F.). (H. J. Tapsell and A. E. Johnson, *Special Report No. 17*, Department of Scientific and Industrial Research (1931).)

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[1] P. G. McVetty, "Working Stresses for High-Temperature Service," *Mechanical Engineering*, March, 1934, p. 149.

A Proposed Method for Interpreting and Correlating Creep Data.

To show the possibilities of such a method, use will be made of creep data in a report by H. J. Tapsell and A. E. Johnson.[1] The work was done at the National Physical Laboratory under the most favorable conditions for securing reliable data. The two materials were carefully heat treated prior to the tests to ensure stability. For the purpose of this analysis, the data in Table I have been taken from Tables II, III and IV of their report.

Following the method previously explained,[2] each creep curve may be replaced, for the purpose of extrapolation, by a straight line having the equation:

$$\epsilon'' = \epsilon_o'' + v_o t$$

where ϵ'' = final plastic strain or creep in inches per inch,
ϵ_o'' = initial plastic strain in inches per inch,
v_o = minimum creep rate in inches per inch per hour, and
t = time in hours.

Since the values of ϵ_o'' and v_o are functions of the applied stress, they may be so plotted as shown in Figs. 2 and 3. It would be very desirable to have tests at lower stresses but in their absence it is permissible to extend the curves to the origin. To facilitate the reading of values of the two variables at low stresses, each curve has been replotted to a larger scale on the same sheet with values indicated along the curves.

By selecting values of ϵ_o'' and v_o from these curves for the desired range of stresses, it is possible to compute the creep at the end of various time intervals. This permits the plotting of the estimated creep at the end of 2 years, 4 years, etc., in a form which represents the effect of time upon the stress-strain curves as shown in Figs. 4 and 5.

These charts give the designer the desired information as to the relations among the variables stress, strain and time for the given temperature. They assume that inflections will not occur within the service life of the material at deformations less than one per cent and that the creep curve at low stresses may be extrapolated along the final tangent. They take into account the initial plastic deformation obtained by extrapolating the final tangent to zero time.

If, for example, the designer wishes to know what stress he may

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[1] H. J. Tapsell and A. E. Johnson, "The Strength at High Temperatures of a Cast and Forged Steel as Used for Turbine Construction," *Special Report No. 17*, Department of Scientific and Industrial Research (London) (1931).

[2] P. G. McVetty, "Working Stresses for High-Temperature Service," *Mechanical Engineering*, March, 1934, p. 149.

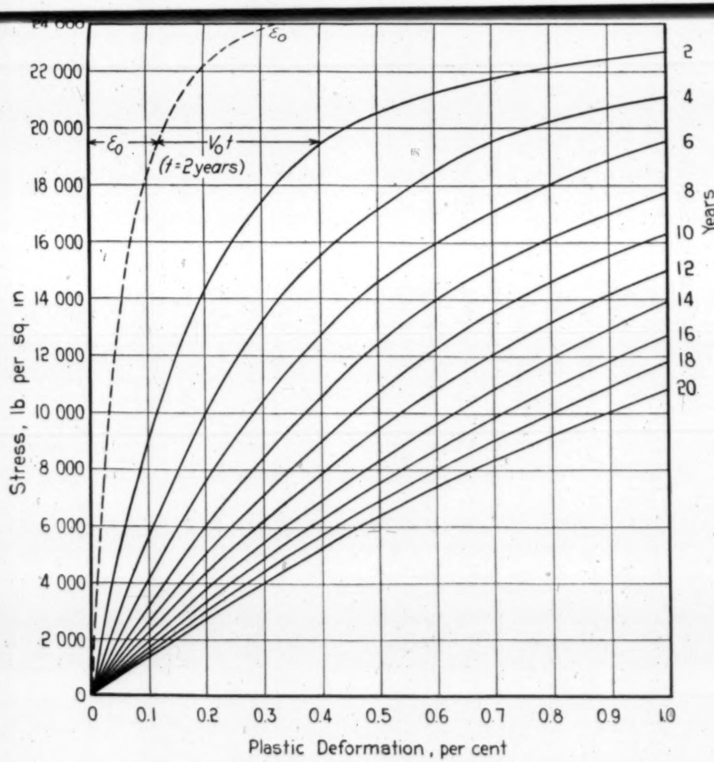

FIG. 4.—Stress-Strain-Time Relations at Constant Temperature.

0.30-per-cent carbon cast steel—400 C. (750 F.). Based on creep tests of H. J. Tapsell and A. E. Johnson, *Special Report No. 17*, Department of Scientific and Industrial Research (1931).

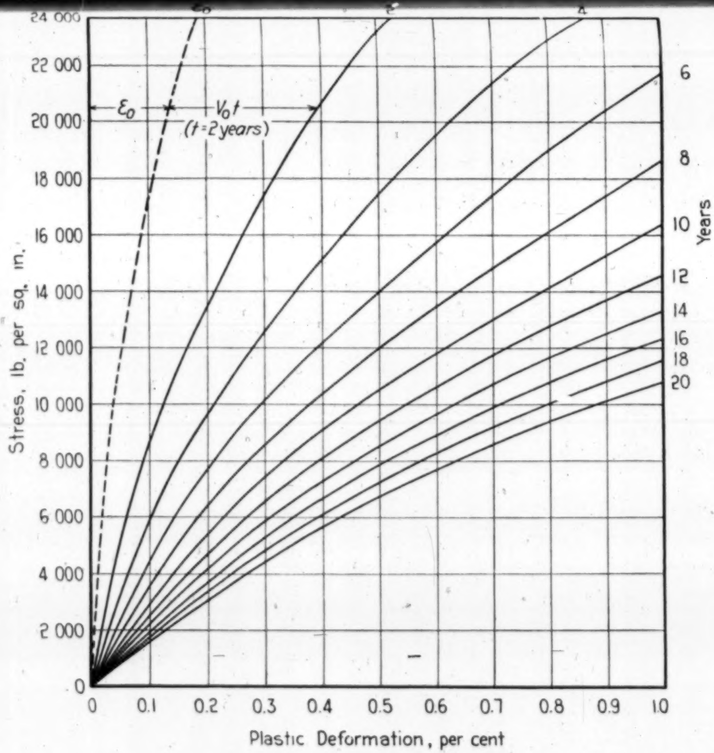

FIG. 5.—Stress-Strain-Time Relations at Constant Temperature.

0.39-per-cent carbon forged steel—400 C. (750 F.). Based on creep tests of H. J. Tapsell and A. E. Johnson, *Special Report No. 17*, Department of Scientific and Industrial Research (1931).

use to keep the plastic deformation within the limit of 0.5 per cent in 10 years, he reads from Fig. 4 for the cast steel a stress of 10,700 lb. per sq. in. and from Fig. 5 for the forged steel a stress of 10,500 lb. per sq. in. Similar charts for other materials allow comparisons on the basis of allowable deformation and expected service life.

This method of comparison is offered as a tentative suggestion. The difficulty in making corrections for the effects of changes in the many variables involved is fully realized. If, however, all available data were reduced to this form and compared, inconsistencies would be revealed and the reliability of all the data improved by a few check tests. It is only by correlation of this kind that the maximum value may be obtained from existing data.

In mapping out any program of creep tests, economic considerations demand that their number and duration be reduced to an absolute minimum. By the method here suggested, it is possible to form some estimate of a new alloy by as few as two creep tests. If this preliminary study is encouraging, the accuracy of the work may be improved by more tests.

CONCLUSIONS

In conclusion, it has been shown that creep tests have very definite requirements and limitations. While objections may be raised to their extrapolation, they are practically useless to the designer unless extrapolated to times representing the expected life in service. Some of the present methods of interpretation have been discussed with particular reference to the importance of structural stability in service. The use of creep rate alone to evaluate a material for a given stress and temperature has been shown to be incomplete. Finally, a method of interpretation has been suggested which offers some advantages over those previously used and indicates a possibility of correlating and making use of the large mass of creep data now available.

DISCUSSION

Mr. H. F. Moore.[1]—Possibly some statements as to the observed creep of lead and lead alloys may be of interest in connection with Mr. McVetty's paper. Lead flows at ordinary room temperatures and the flow is relatively rapid, so that simple apparatus can be used to study the phenomenon of flow. However, in connection with the study of lead it must be remembered that there is always the possibility of recrystallization which may affect quantitative results.

For some years there has been going on in the Materials Testing Laboratory at the University of Illinois a study of creep of lead and lead alloys, and there have been obtained some test results from tests showing a continuous flow for over $2\frac{1}{2}$ years.

There is some indication that, at a given temperature, the rate of creep of lead alloys tends to diminish, not in a straight-line relation with stress, but in a relation which seems to give some indication that as stress diminishes the stress-creep line approaches a horizontal asymptote. The stress-creep curve looks something like a left-handed *S–N* diagram for a fatigue test, although the limiting stress is not nearly so clearly defined. So far we have found no stress, even at 0 C. (32 F.), so low that *no* creep takes place in lead.

Viewed through the metallographic microscope the creep of lead presents interesting features. A specimen is prepared by slicing lead on a microtome. A given small area is photographed with a magnification of about 100. This photograph shows merely scratches left by the microtome knife. After a few score hours of creep the outlines of the crystalline grains begin to appear, and soon become so distinct that they give the impression of cracks. However, by rotating the illumination of the microscope the apparent "cracks" are seen to be due to unevenness of surface at grain boundaries. In the meanwhile the microtome scratch lines are seen to change their direction slightly in certain grains indicating slight rotation of grain. Actual cracks appear only when necking down of the specimen begins and final tensile fracture is approaching. These cracks are intercrystalline, as are cracks in lead alloys produced by repeated stress.

A few tests have been made on single crystals of lead but so far the rate of creep seems to be affected by the orientation of the possible slip planes with reference to the axis of pull.

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[1] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

Mr. McVetty spoke of the embrittlement as shown after a long period under steady load; the same embrittlement has been noticed in certain lead alloys under a long, steady load, where, for a time, the elongation increased with the amount of time required to fracture, then reached a maximum and decreased. The fracture looked quite different from a short-time fracture, and there was much less reduction of area.

Most of all I should like to commend Mr. McVetty's contribution to the philosophy of interpretation of creep test results. I am especially struck by his beautiful phrase to the effect that extrapolation is a necessary evil and it may be useful if it is on the safe side. I think that is excellent *engineering* philosophy, though not so good as *scientific* philosophy. I wish also to emphasize Mr. McVetty's suggestion that while we have done a good deal of cooperative work in *making* tests we may well now turn to cooperative work in the *interpretation* of tests.

MR. T. McL. JASPER[1] (*by letter*).—I have to admire the ingenuity Mr. McVetty has used to bring some semblance of logical reasoning into the question of creep testing. The results shown by the method of creep testing in the past have been very unsatisfactory to the engineer and the user of metals. This, I believe, has been largely due to the fact that the results obtained by several investigators using creep methods have varied for similar steels over a wide range of values. No doubt this is due largely to the fact that the precision of measurements and control of temperature necessary to produce consistent results is of an order which may not be applied with ordinary laboratory equipment or with the methods of temperature control usually available. It would accordingly seem to the writer that the creep method of testing for values at elevated temperatures is not the best.

By creep testing is meant in this discussion the obtaining of values defined as 1 per cent creep in 10,000 hr. or 1 per cent creep in 100,000 hr. as a certain criterion of strength. These values have been given the term "creep rate."

There are two very difficult influences to overcome in creep testing which are, first, the effect of initial stresses in the steel being tested, and second, the effect of reducing the cross-sectional area of the test sample by oxidation or corrosion during testing. Both of the above influences would be of minor importance if it were not for the fact that the instruments necessary to use in measuring the stretch of the metal need to be of a sensitivity of the order of 0.000001 in. and if the number of hours indicated by the rates did not represent such long periods of time.

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[1] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

With reference to the first influence, reference is made to some very careful creep testing work done by Kanter and Spring and presented before this Society in 1928.[1] Figure 15 of that paper shows the tests on a single steel. These tests are on an 0.35 per cent carbon steel in the annealed, normalized and quenched condition and are made at 425 C. (800 F.), and the stresses imposed on the test samples are identical. Above about 500 hr. the slope of each curve is practically identical. The initial slopes of the various curves differ, due probably to differences in initial stress conditions. It is not desired to go into the details of such effects as are caused by initial stresses but the mechanics of this was discussed by the writer in the *Transactions* of the Institute of Engineers and Shipbuilders in Scotland, Vol. LXXI, Part VI, page 592 (1928).

Referring to time-strain creep curves, these are so complex for various steels that it is doubtful in the writer's mind whether at high temperatures, say 480 to 540 C. (900 to 1000 F.) the values obtained by creep methods of testing are not affected for a considerably longer time than is intimated above. To bear this out reference is made to Fig. 16 of the paper by Kanter and Spring referred to above which shows some of the stronger steels with lower values of "creep strength" than the weaker steels.

The second point is the small depth of oxidation or corrosion necessary to reduce the area of the test sample a few per cent during the test and since the rate deals with a very small per cent in a long time the rate of reduction of cross-sectional area bears in an important manner on the slope of the curve which results from the time strain rate. If, for instance, in 11 years or 100,000 hr. this reduction of cross-sections due to oxidation is, say 4 per cent, then the effect on the curve can be nearly the whole of the slope plotted as 1 per cent. This, of course, is more effective on carbon steels because oxidation or corrosion rates for such steels are relatively high. It also depends on the actual strain imposed on the test sample. In this connection it is appropriate to point out that the modulus of elasticity for steel at high temperatures is very much reduced over that which is effective at ordinary temperatures.

To present an idea as to how much depth of oxidation would be necessary to reduce the area of a $\frac{1}{2}$-in. test specimen 4 per cent, the value is 0.005 in. This seems a small amount to occur in 11 years or even one tenth of this time at high temperature provided oxidation or corrosion is available. It must be obvious that as the cross-sectional area decreases the specimen lengthens elastically when the load is kept constant.

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[1] J. J. Kanter and L. W. Spring, "'Long-time' or 'Flow' Tests of Carbon Steels at Various Temperatures with Particular Reference to Stresses Below the Proportional Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 80 (1928).

The writer's lack of enthusiasm for the creep method of testing is undoubtedly engendered by the belief that a very much less tedious method of obtaining reliable results on the strength of metals at elevated temperatures is available. Methods which have been used for a considerable period by the writer are offered as a substitute for the creep method of testing metals at elevated temperatures. They are described in the April, 1933, issue of the *Refiner*.

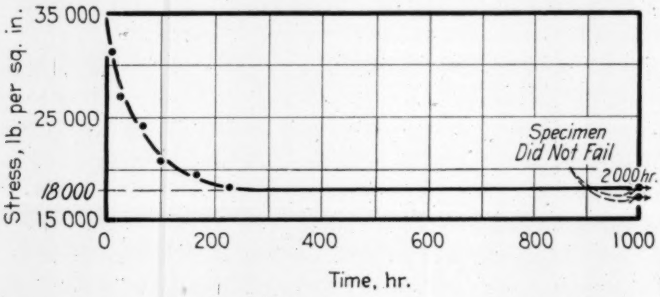

FIG. 1.—Typical Stress-Time Relation for 0.20 to 0.30 per cent Carbon Steel when Tested at 480 C. (900 F.)

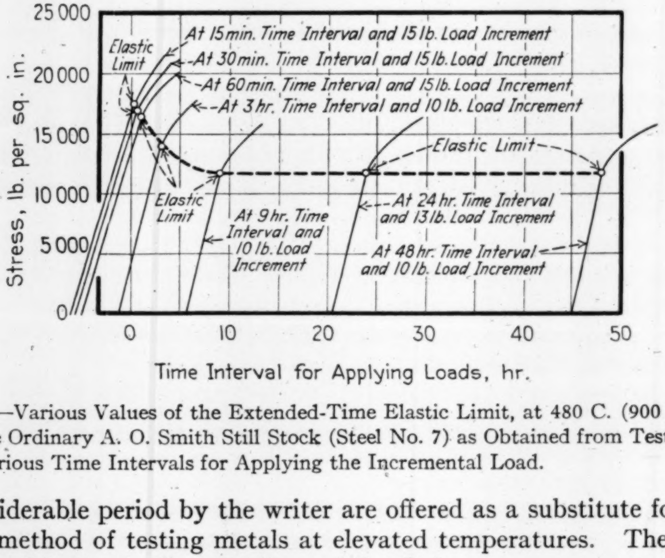

FIG. 2.—Various Values of the Extended-Time Elastic Limit, at 480 C. (900 F.), of the Ordinary A. O. Smith Still Stock (Steel No. 7) as Obtained from Tests with Various Time Intervals for Applying the Incremental Load.

The method used in obtaining the long-time ultimate strength of steel at high temperatures was adopted first by H. J. French when he was associated with the National Bureau of Standards. It is indicated by the accompanying Fig. 1 which is self-explanatory and can be applied with the generally accepted creep-testing equipment. It consists of running an endurance test with the steel tested at various stresses until a stress sufficiently low is obtained at which failure does not occur for a very long period of time.

The method of obtaining the long-time elastic strength of steel at high temperatures is indicated in Fig. 2. Each stress-strain diagram represents a different rate of loading the test specimen. The value of the elastic strength for each rate of loading is plotted along the time axis and represents the time which each increment of load was allowed to exert itself before another increment of load was added. The elastic strength values so obtained are represented along one ordinate and the time for each increment of loading along the other ordinate. By joining the elastic strengths so plotted a curve resembling an endurance curve is obtained which approaches a constant value. This method of obtaining elastic strengths at elevated temperatures, used since 1928, has proved to be reproducible without instruments of great sensitivity or precision and with the ordinary degree of care used in laboratory work.

The question of temperature control and of sensitivity of measuring equipment need not be of the same degree of precision as the creep method of testing requires to produce values within a very small range when different operators use the methods outlined above. The writer uses protection against excessive oxidation and temperature control equipment which is easily obtainable.

MR. P. G. MCVETTY[1] (*author's closure by letter*).—We are indebted to Mr. Moore for his contribution to our knowledge of flow phenomena as revealed by creep tests of lead and lead alloys at normal temperature. While a quantitative comparison may not be allowable, these tests give valuable indications of the probable action of other crystalline materials, such as heat-resisting metals and alloys at high temperatures. The analogy may be carried further by the use of "homologous temperatures" as suggested by Ludwik.[2] It is particularly significant that no stress has been found low enough to prevent creep of lead. This is in agreement with the assumption that the high-temperature creep rate cannot be zero for a finite value of stress.

The rotation of grains observed under the microscope indicates an adjustment in crystals unfavorably situated with respect to the direction of the applied stress. These adjustments may account for the change of deformation rate in the early stage of a creep test.

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[1] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] A. Nadai, "Plasticity," p. 275, McGraw-Hill Book Co., Inc. (1931).

Tests of single crystals to determine the effect of orientation upon creep rate may indicate a method of improving creep properties. This possibility leads us to hope that Mr. Moore will soon publish the results of this very interesting research.

Mr. Jasper's substitute for the creep method of testing has many points in its favor for a large number of high-temperature applications. It is seldom desirable to use creep tests unless the allowable deformation is closely limited. In many applications, however, the limiting deformation is fixed by the clearance between moving and stationary parts. The stress must be chosen to give a satisfactory service life without exceeding the prescribed deformation limit. In such cases it is doubtful if the designer would accept the proposed method of determining the elastic limit unless creep tests confirmed the assumption of zero creep at stresses below this limit. A comparative test of this kind would allow a better evaluation of the proposed method.

In reference to the effects of oxidation or corrosion, Mr. Jasper mentions an important point which must always be considered. Allowance for loss of cross-section in service may be made by computing stress on the basis of estimated final area. The elastic deformation which accompanies the gradual stress increase due to loss of cross-section during a constant load test is frequently neglected. A reasonably accurate estimate of its value is easily made and applied as a correction to the observed creep readings. For this purpose it is necessary to determine the modulus of elasticity at the temperature of the test.

Considerable attention has been directed toward the standardization of the details of creep testing so as to make the results comparable. The analysis and correlation of creep data and their presentation in convenient form for design use represent another problem of great practical importance. When this has been accomplished, we may expect a better evaluation of the various accelerated tests which have been proposed.

OBSERVATIONS ON EFFECT OF SURFACE FINISH ON THE INITIAL CORROSION OF STEEL UNDER WATER[1]

By L. J. Waldron[2] and E. C. Groesbeck[2]

Synopsis

It is well known that the surface of certain metals is coated with an oxide film which is oftentimes invisible if it is closely adherent to the metal. This coating may exert a protective effect on the underlying metal in a corrosion test so long as it remains intact, or it may cause irregularities in the results, especially if there are discontinuities present in the oxide film due to expansion, contraction, bending, scratching, etc.

This investigation was undertaken to ascertain what effect, if any, different surface finishes may have on the results obtained in the initial stages of any corrosion test. An apparatus of the gravity flow type was used for the tests with the water (Washington city) circulated by a pump. The water flowed vertically down past the specimens at various velocities ranging from 5 to 225 ft. per min.

The points studied were: (*a*) The effect of variations in the surface finish on the loss in weight of specimens with and without an oxide film at low and high velocities of flow; (*b*) the time of exposure to the air during which an oxide film forms on coarsely ground specimens prior to test; and (*c*) the relation between duration of immersion (up to 28 days) at various velocities of flow and the loss in weight of specimens with or without an oxide film.

The experimental data are presented in three diagrams and one photograph. In general, the type of finish is not an important consideration at low velocities of flow, but is at high velocities. The character of the atmosphere and the length of time the specimens are exposed prior to test may have an effect on the test results.

Introduction

The existence of air-formed oxide films on the surface of metals, such as iron, steel, copper, zinc and aluminum, has been established by various investigators.[3] Coatings of this kind usually are invisible if they are thin and closely adherent to the underlying metal. They may protect the metal from further oxidation by preventing the inward diffusion of more oxygen. On the other hand, they may cause irregularities in the results of corrosion tests, especially if there are discontinuities in the oxide film. Such defects may be: (*a*) breaks, caused by expansion and contraction of the metal or by its being bent, scratched, etc; (*b*) "weak spots" which may conceivably be produced by inclusions in the metal at the surface; or (*c*) a break-down of the oxide film in certain solutions.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Assistant Metallurgist, and Metallurgist, respectively, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[3] See the various references given in Chapter III of E. S. Hedges' book, "Protective Films on Metals" (published in 1932 by D. Van Nostrand Co., New York City, and also by Chapman and Hall Ltd., London, England), and also the several references in the paper by S. C. Britton and U. R. Evans, "The Distribution of Corrosion," *Transactions*, Am. Electrochemical Soc., Vol. LXI, p. 441 (1932).

The present contribution is offered for the purpose of calling attention to the importance of giving proper consideration to the surface finish of specimens used in short-time "under water" corrosion tests.

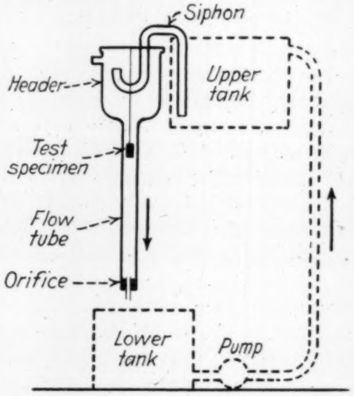

FIG. 1.—Diagram Showing the Principal Features of the Apparatus.

APPARATUS

During the tests, water was circulated by a rotary pump from one tank to another at a higher level and from this vertically down through a series of testing units. All parts in contact with the water were of glass or rubber. The apparatus was so designed that the number of units could be varied, the maximum capacity of the system being six units for the velocities used.

The general details of one of these units are shown (in solid lines) in Fig. 1. A glass siphon extended from near the bottom of the upper tank to a glass header. The upturned outer end of the siphon was approximately level with the outlet which maintained a constant level in the header. This arrangement facilitated the separation of air bubbles from the water. The overflow returned down to the lower tank. A glass tube, about 36 in. (90 cm.) long and about 1 in.

(2.5 cm.) in diameter, attached to the lower end of the header by a rubber connection, served as the flow tube through which the rate of flow was controlled by an orifice at the bottom. The water passed through the flow tube directly into the lower tank, which was provided with an outlet to permit any excess water to flow into the drain. The specimens were suspended within the flow tube by cotton thread through a hole in the specimen and attached to a glass rod placed across the top of the header. Each specimen, with a hole 0.03 in. (0.08 mm.) in diameter at each end, was tapered so as to reduce as much as possible any turbulent effects set up in the water stream by the specimens.

TESTING METHOD

Six units, having flow rates from 5 to 225 ft. per min. were used. For the higher velocities it was necessary to replace the 1-in. tubes with others of smaller diameters.

Washington city water,[1] having a pH value of 7.6 to 7.8 and a dissolved oxygen content of approximately 5 cu. cm. per liter, was used as the corroding medium. The water was kept saturated with air during the test and its temperature was automatically maintained at 30 ± 0.5 C. Fresh water was added to the system at the rate of about 10 to 15 gal. per hr., the capacity of system being about 45 gal.

The material used for this study was No. 22 gage open-hearth steel of the following composition: carbon, 0.02 per cent; manganese, 0.38 per cent; silicon, 0.006 per cent; sulfur, 0.023 per cent; phosphorus, 0.006 per cent; copper, 0.044 per cent. Rectangular specimens about 2 in. long and 0.75 in. wide (5 by 1.9 cm.), were used.

All specimens were given a preliminary treatment on a belt grinder to remove scale and other surface defects. They were then ground by hand on No. 1 G emery paper in the same direction as on the belt grinder; this finish is designated as "coarse." Whether the oxide film was to be retained or removed by pickling, the specimens were cleaned with alcohol, benzol and ether, measured and weighed. Those specimens which were to be pickled were treated for a few minutes in a 10 per cent hydrochloric acid solution containing an inhibitor, thoroughly washed in water and immediately put into the test. The specimens with the oxide film left on were allowed to stand in a desiccator for definite periods of time, prior to testing, to permit further development of the oxide film. Other specimens to be given a high polish (designated as "fine") were ground on several abrasive papers, following the No. 1 G emery paper, and finished on 0000 emery paper. In each operation the specimen was so held that the scratches produced were at right angles to those formed by the previous abrasive. The specimens were then cleaned, measured and weighed as above. Any variations from this general procedure of preparation are dealt with in the next two sections.

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[1] The composition of the water was quite similar to that reported in Table IV.—Analysis of Filtered Washington City Water, Report of Subcommittee V of Committee A-5 on Corrosion of Iron and Steel, see *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part I, p. 161 (1928).

Previous to reweighing after test, all specimens were freed from any adhering rust by a momentary pickling in the 10 per cent hydrochloric acid solution containing an inhibitor.

RESULTS OF TESTS

Neither the kind of wash water (tap or distilled), the temperature, the dissolved oxygen content of the water nor the method employed for washing the specimens after the pickling prior to testing exerted any appreciable influence upon the weight losses obtained, provided the specimens were not allowed to dry.

TABLE I.—EFFECT OF SURFACE FINISH ON THE LOSS IN WEIGHT OF SPECIMENS WITH NO OXIDE FILM.

Finish	Velocity of Flow, ft. per min.	Number of Specimens	Average Corrosion Loss, g. per sq. dm.
Coarse	11	11	0.26
Coarse, retested[a]	11	5	0.25
Coarse	225	30	0.57
Coarse, retested[a]	225	2	0.60
Coarse, modified[b]	225	8	0.59
Fine	225	2	0.64

[a] This finish was obtained by a short repickling (30 sec.) of coarse-finish specimens which had been tested previously and consequently were in a more or less corroded condition when put into test again.

[b] The specimens were ground on No. 1 G emery paper in a direction at right angles to that used on the belt grinder until the scratches produced by the previous abrasive were removed.

Preliminary Tests on the Effect of Surface Finish on Loss in Weight of Specimens Having No Oxide Film:

Table I shows that variations in surface finish had no appreciable effect on the loss in weight of those specimens that had the oxide film removed by pickling, during a 48-hr. test, provided that the velocity of flow was maintained constant. However, a distinct increase in the corrosion rate was produced when the velocity of flow was raised.

Effect of Surface Finish on Corrosion of Specimens Having an Oxide Film:

The effect of variations in the surface finish (produced by different abrasives) on the loss in weight of unpickled specimens is shown in

Fig. 2. The specimens were kept in a desiccator for 30 to 40 min. before being tested for 48 hr. in water flowing at rates of 11 and 225 ft. per min., respectively. The appearance of fine-finished specimens (600 grains per inch) after exposure at the low and high velocities, is shown in Fig. 3, specimens *A* and *B*.

At low velocity of flow, changes in the surface finish had practically no effect on the corrosion losses, until very fine finishes were used, when there was a distinct, though not large, lowering in the corrosion losses. At the high velocity, a marked change in the corrosion losses was produced by a slight change in the particles size of the coarse abrasives, but finishes obtained with abrasives of medium and fine particle size gave low and quite constant losses.

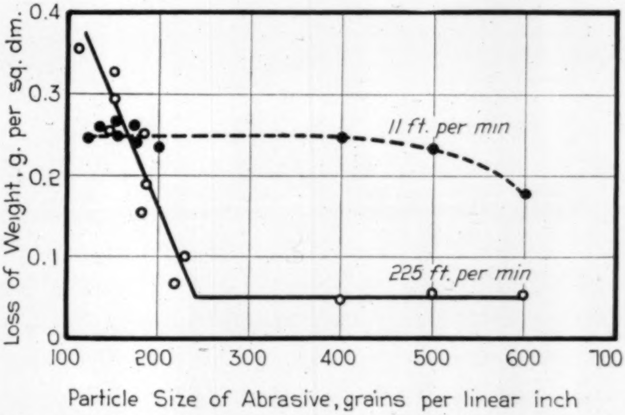

FIG. 2.—**Effect of Surface Finish on the Loss in Weight of Unpickled Specimens.**

The values given for the particle size of abrasive for the various abrasives are only approximate. Each point represents at least two specimens.

Effect of Time During Which Unpickled Specimens Are Exposed Prior to Test:

Specimens with the coarse finish were used for determining what effect, if any, the period of exposure to the air, prior to test, would have on the corrosion rate. After grinding, the specimens were exposed to the dry atmosphere in a desiccator at room temperature (18 to 22 C.) for various periods up to 24 hr. before being tested for 48 hr. in water flowing at 225 ft. per min. In Fig. 4 the points at zero time are for pickled coarse-finish specimens. The appearance of specimens, which had previously been exposed to the dry air for 1 hr., after test is shown in Fig. 3, specimens *C* and *D*.

The exposure of coarse-finish specimens to the dry air for about an hour or less produced a marked lowering in the corrosion losses. Practically no further change in the corrosion losses occurred when the specimens were exposed for longer periods up to 24 hr. The corrosion losses were markedly lower for specimens having a slightly smoother finish (curve *B*), which was obtained by modifying the grinding procedure (Table I, footnote *b*).

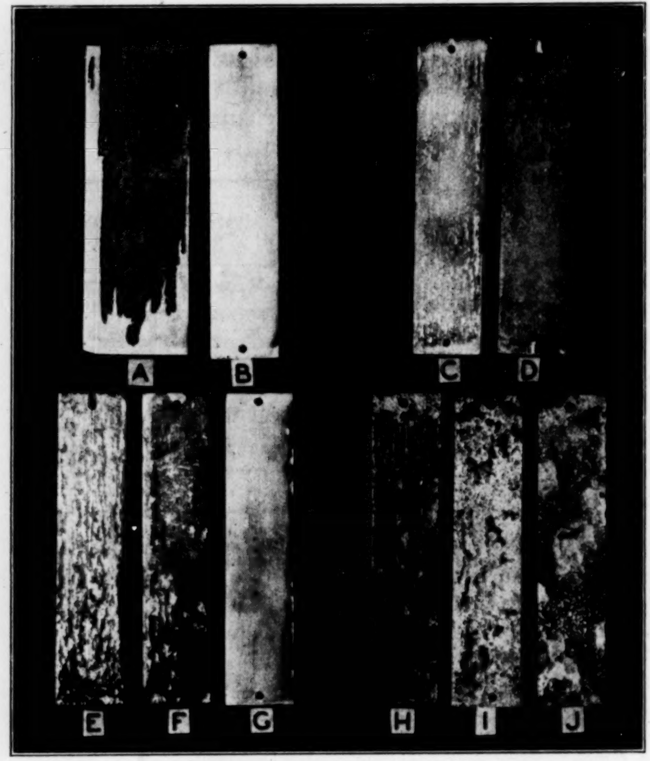

FIG. 3.—Appearance of Specimens After Test.

Flow rates of 11 and 225 ft. per min. were used for specimens A and B, respectively. Specimens A and B had a fine finish. Specimens C to J were corroded at a flow rate of 225 ft. per min.; specimens C and D, which had a coarse finish, were exposed to the air in a desiccator for 1 hr. prior to test. Specimens E, F and G were corroded for 6 days and specimens H, I and J for 28 days; see Fig. 5 for the surface finish of these specimens in the order as indicated.

It was found that at a very low velocity (5 ft. per min.) the length of exposure to dry air has no significant effect on the corrosion rate, that is, a constant loss in weight was obtained for exposures lasting from 0 to 50 hr.

Results obtained from numerous experiments showed that the oxide film formed on specimens when exposed to the laboratory air exerted much less protective effect than did that formed in a desiccator. It appears that the protection given by the oxide film is dependent upon its degree of hydration, that is, the humidity under which it was formed.

Relation Between Surface Finish and Duration of Corrosion Period:

Figure 5 shows the relation between the time of immersion (up to 28 days) and the corrosion losses obtained for specimens having a coarse finish (with or without oxide film), and a fine finish, at low and high velocities of flow. The test periods were 2, 6, and 28 days. The appearance of specimens immersed for 6 and 28 days at the high velocity is shown in Fig. 3, specimens *E* to *J*.

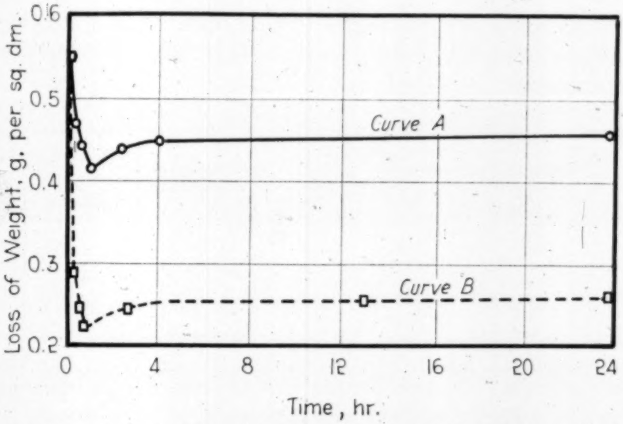

FIG. 4.—Effect of Period of Exposure to the Air Prior to Test on Specimens Having a Coarse Finish, Using 225 ft. per min. for the Velocity of Flow.

Curve A; Ground in same direction as on belt grinder. Curve B; Ground in direction at right angles to A. Each point represents at least two specimens.

The corrosion losses obtained at the low velocity with the different finishes were substantially the same in 2 days. However, as the corrosion period was increased, the loss became the greatest with the pickled coarse-finish specimens and the least with the fine-finish specimens. The oxide film probably gives some protection during the early stages of immersion. But the coat of corrosion products which forms and in time spreads over the surface of the specimens probably helps to lessen the loss in weight. The results were different when a high velocity of flow was employed. The corrosion loss for the pickled coarse-finish specimens was initially greater than that for the fine-finish specimens, but it decreased as the time of immersion was increased, and after 28 days the losses were less than those for the fine-finish specimens. The loss in weight of the unpickled coarse-finish specimens increased steadily with time, similarly to that for the fine-finish specimens, but with somewhat greater losses.

Reproducibility of Results:

An analysis of the test data by the procedure recommended by the Society[1] showed that good control was secured when the flow rate was low (5 and 11 ft. per min.). But when the flow became more turbulent, as at higher velocities, the control was not so good, especially in the case of specimens bearing an oxide film. The following shows the variations from the average values that were obtained with the pickled specimens:

Velocity of Flow, ft. per min.	Number of Observations	Amount of Deviation, per cent	
		Maximum	Average
5	11	5.3	3.3
11	13	4.3	1.6
18	11	8.9	4.2
31	11	9.8	3.8
63	11	12.0	6.8
225	26	12.5	7.2

The reproducibility of the results for the oxide-film specimens decreased as the velocity of flow was increased.

DISCUSSION OF DATA

The foregoing results indicate that the presence of an oxide film on the specimens prior to immersion substantially lowers the loss in weight. The magnitude of this restraining effect depends upon the character of the oxide film, velocity of flow of water, duration of the corrosion period, and composition of water. The character of the oxide film is dependent upon the surface finish of the metal, the atmosphere to which the specimen is subjected prior to immersion and duration of exposure to that atmosphere. Passano and Nagley[2] have expressed the belief that non-uniformity in distribution of corrosion is associated with air-formed oxide films.

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[1] Manual on Presentation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 451 (1933). See also W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., New York City (1931).

[2] R. F. Passano and F. R. Nagley, "Consistent Data Showing the Influence of Water Velocity and Time on the Corrosion of Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 396 (1933).

Some of the tests reported above were repeated in distilled water having a pH value of 6.0 to 6.5 instead of tap water. The results obtained indicated that the oxide films formed in dry air in a desiccator, previous to immersion in the water, have a far less important effect in distilled water than in tap water, even at a high velocity (225 ft. per min.).

The results of an experiment, in which highly polished specimens were pickled to remove any oxide film present and were then exposed to the dry air (desiccator) to form the oxide film again, showed that substantially no protection against corrosion was given by the renewed oxide film. This indicates that a "flowed" metallic surface may have an influence on the formation of the oxide film.

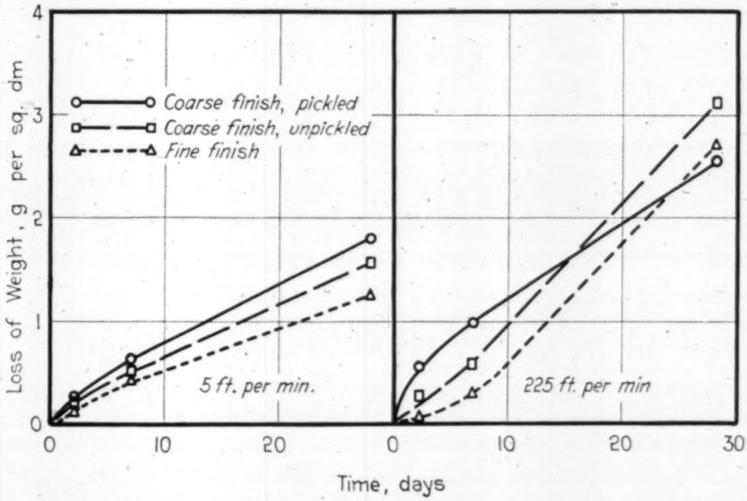

FIG. 5.—Relation Between Duration of Corrosion Period and Loss in Weight of Selected Finishes.

Each point represents 2 to 30 specimens tested for 2 days, 4 specimens tested for 6 days and 2 or more specimens tested for 28 days.

The results of the tests indicate that, in general, the presence of an oxide film on the specimens does not have any marked influence on the corrosion losses at low velocities of flow, but that it does at relatively high velocities.

These results were secured for relatively short periods of immersion, and may not have any relation to the effects obtained during longer periods such as in service.

DISCUSSION

Mr. R. F. Passano.[1]—The authors used rectangular specimens 2 in. long and 0.75 in. wide in their experiments, which specimens were cut from a No. 22 gage sheet of steel. Counting the four edges, the specimens have a surface area of roughly 20 sq. cm. The losses were reported in grams per square decimeter, which is equivalent to multiplying the loss per specimen by 5. If the loss of weight per infinitesimal area is a random function of the total area from which specimens of any given area are cut, the standard deviation of the losses for a specimen with an area of 100 sq. cm. (1 sq. dm.) will be $\sqrt{5}$ times the corresponding standard deviation for specimens with an area of 20 sq. cm. In case the loss of weight from one element of area is correlated with that from another, it is necessary to know the nature of the correlation before one can determine the relation between the standard deviations of losses from specimens of different areas. It is almost certain that this complication exists in corrosion investigations. There is thus good reason to report data on the basis of the test specimen without conversion to an arbitrary unit until such time as there are adequate data on known sampling methods to calculate the relation between the statistics obtained from specimens having different areas.

I have chosen to discuss this matter in terms of the standard deviation because the Society's Committee on Interpretation and Presentation of Data recommended last year that the essential information in a series of observations was best conveyed in cases of this sort by the presentation of the average, the standard deviation, and the number of observations. The authors chose to give information on the distribution of their losses in terms of the average deviation which is a less efficient measure of dispersion than the standard deviation, but there is a similar complication in conversion no matter which statistic of dispersion is chosen.

The authors have advised me that their table showing the amount of deviation experienced at velocities between 5 and 225 ft. per min. was based on losses from coarse-finished, pickled specimens exposed for 2 days at each velocity. I am of opinion that (1) when the conditions are controlled in a statistical sense, the deviations depend principally on the loss of weight and not on the conditions used to produce the loss; and (2) when the conditions are not controlled in a statistical sense, the observed deviations are a reflection of the relative completeness of attack from specimen to specimen. Considering the problem from this angle, it would be desirable to have available for the data covered by this paper:

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[1] Research Engineer, Research Laboratories, The American Rolling Mill Co., Middletown, Ohio.

(*a*) Evidence of statistical control,

(*b*) Knowledge of the completeness of the attack, and

(*c*) The average loss associated with each of the reported deviations.

I have estimated from Fig. 5 that the loss from the coarse-finished, pickled surface at 5 ft. per min. was 275 mg. per sq. dm. and from the same kind of surface at 225 ft. per min. was 550 mg. per sq. dm. These values are equivalent to losses of approximately 55 and 110 mg. from specimens with an area of 20 sq. cm. For 11 specimens at 5 ft. per min. the average deviation is given as 3.3 per cent (1.65 mg.), and for 26 specimens at 225 ft. per min. the average deviation is given as 7.2 per cent (7.92 mg.). Presuming that the standard deviation is equivalent to $\sqrt{\frac{\pi}{2}}$ times the average deviation, I have calculated the standard deviation, corrected it for sample size and compiled the following table:

TIME, DAYS	VELOCITY, M. PER MIN.	NUMBER OF OBSERVATIONS	AVERAGE LOSS, $\bar{X}$, MG. PER 20 SQ. CM.	STANDARD DEVIATION, σ', MG. PER 20 SQ. CM.
2	1.5[a]	11	55	2.3
2	67.5[b]	26	110	10.3

[a] 5 ft. per min.
[b] 225 ft. per min.

With these data, I should like to compare some that Nagley and I reported last year:[1]

TIME, DAYS	VELOCITY, M. PER MIN.	NUMBER OF OBSERVATIONS	AVERAGE LOSS, $\bar{X}$, MG. PER 20 SQ. CM.	STANDARD DEVIATION, σ', MG. PER 20 SQ. CM.
2	1	25	54.2	2.6
3	5	25	117.7	5.1

The values obtained by Waldron and Groesbeck represent measurements made in a soft water with a pH value of 7.7, and a dissolved oxygen content of 7.1 p.p.m. at a temperature of 30 C. Those obtained by Nagley and me represent measurements made in a hard water with a pH value of 7.5 and a dissolved oxygen content of 10.2 p.p.m. at a temperature of 14.9 C. At the lower velocity shown in each of these two tables (where the loss was about 55 mg.

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[1] R. F. Passano and F. R. Nagley, "Consistent Data Showing the Influence of Water Velocity and Time on the Corrosion of Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 387 (1933).

per specimen), the estimated standard deviation of the distribution was practically the same. At the higher velocity shown in each of the two tables, the loss of weight is approximately the same, but the estimated standard deviations are quite different, the one shown by Waldron and Groesbeck being larger than that which Nagley and I observed.

In the course of their paper, Waldron and Groesbeck specifically say nothing about the completeness with which their specimens were attacked unless it be by implication. They investigated the effect of abrasive grain size and of oxide films. It was from such a study that Nagley and I concluded: "the method of preparing the surface for test is unimportant *provided the specimen corrodes all over.*" Specimens prepared by different methods do not corrode all over with equal frequency. The average loss and standard deviation reported by us were based on specimens which were completely attacked over their whole surface from the beginning of the test: I presume from what Waldron and Groesbeck say and my knowledge of the subject that their losses and deviations are based on specimens which were not attacked all over. Thus I am inclined to explain the difference between the standard deviation which they found for a loss of 110 mg. and that which we found for approximately the same weight loss on the presumption that the completeness of their attack was variable, whereas ours was constant. I recognize that there is extreme difficulty in preparing specimens so they will corrode all over at high velocities. When one establishes a policy that the specimens must corrode all over if any loss of weight measurement is to be

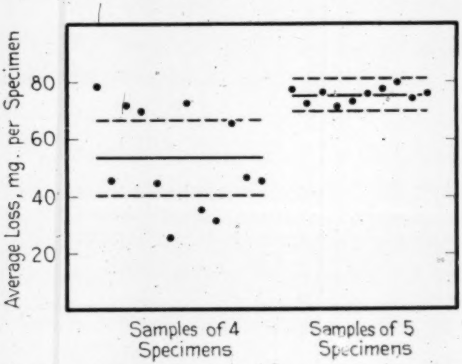

FIG. 1.—Graphical Presentation of Data from Two Series of Underwater Experiments, the One Involving Specimens that Were Incompletely Attacked and the Other Specimens that Were Completely Attacked.

recorded, it is quite disheartening to realize that the method one has been using to prepare the surface is not satisfactory for the bulk of the specimens started at a new, higher velocity,[1] and to admit that the loss of weight observed depends on the surface finish.[2]

In the accompanying Fig. 1 I have shown graphically the result of two series of experiments conducted in Central Works tap water at a velocity of 1 m. per min. for 3 days. The data on the left were obtained on samples of 4 specimens and the data on the right were obtained on samples of 5 specimens. The specimens in the 12 samples of size 4 shown on the left corroded varying amounts, practically from 0 to 100 per cent of the area of the specimen, whereas the 10 samples of size 5 corroded completely.

Neglecting the influence of the change in sample size which is relatively small in comparison to the effect which is now being discussed, it will be noted that the variation among the samples which corroded only partially is enormous compared to that observed on samples which corroded completely. The variation in the case of the incompletely attacked specimens is greater than should be left to chance, whereas the data available would indicate that when the specimens are completely attacked, the variation is not greater than should be left to chance.[3] I know it to be a fact from observation of the various samples of 4 specimens that those showing the highest weight loss in the series were nearly completely attacked while those showing the lowest weight loss were practically unattacked. I have included this figure in this discussion to point out the information which is given by a test for statistical control and the effect which completeness of attack has on the average loss and on the amount of deviation. The average loss from the 48 specimens which were partially attacked was about 55 mg., whereas the average loss from the 50 specimens which were attacked completely was about 75 mg.

I am interested in corrosion-time data because I believe they convey more information than is conveyed by the loss of weight at some arbitrary time interval. The corrosion-time relationships which Waldron and Groesbeck show in their Fig. 5 are much closer to straight lines over the interval investigated than those which Nagley and I got in hard water. I think this was to be expected and may be explained partially by the fact that the rust formed in hard water contains calcium carbonate. When one undertakes an investigation of a corrosion-time relationship under some particular condition, it is difficult to know where to stop and how many intervals should be investigated between the place at which one begins and the place at which one stops. Nagley and I thought we should make loss measurements at no less than six time intervals (preferably more, of course), that the data representing each time interval should show statistical control, and that the curve alleged to represent the relationship between loss of weight and time should not miss any point by an amount greater than should be left to chance. The number of observations which we made at each time interval was between 10 and 50. We know that even though the values were statistically controlled, the error of the "*n*" observations at each time interval is appreciable. If one takes this error into consideration, there is no need for the curve alleged to show the relationship between loss of weight and time to pass through every point. I believe that some of the curves shown by Waldron and Groesbeck could have been drawn more nearly as straight lines than they have shown them, but this I cannot determine definitely without the basic information which I have indicated as being so important.

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[1] One might have to start 100 specimens in order to find 10 that corroded all over, a 90 per cent rejection.

[2] When a condition is found for which the adopted method of preparing the surface is not suitable, one would be required to repeat his whole program up to that point with the specimens prepared by the new method.

[3] A question of whether the average loss from the samples lies within the limits established by the over-all average plus and minus three times the standard deviation for averages. See W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., New York City (1931).

MESSRS L. J. WALDRON[1] AND E. C. GROESBECK[1] (*authors' closure by letter*).—A statistical study of the test data was made although it was realized that the limited time available had not produced sufficient data to be well adapted for such study. A lack of space did not permit the incorporation of these results in the paper. However, the degree of reproducibility furnished by the authors' test data is indicated in the table given on page 130 of the paper.

It seems to the authors that there is much room for exercise of judgment in attempts to apply statistical analysis in the investigation of corrosion phenomena. The authors believe that many valuable studies of corrosion would be excluded from consideration if all corrosion data must fall within arbitrarily selected limits of scatter.

Borgmann and Evans[2] have pointed out that it is wrong to avoid the study of cases where the scatter is large if the poor reproducibility is not due to insufficient control of external conditions. They add that "'high-scatter' phenomena include many subjects of great practical importance and others of great unique theoretical interest."

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[1] Assistant Metallurgist, and Metallurgist, respectively, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[2] See the section on the cause of poor reproducibility in the paper by C. W. Borgmann and U. R. Evans, "The Corrosion of Zinc in Chloride Solutions," *Transactions*, Am. Electrochemical Soc., Vol. LXV, p. 266 (1934).

It has been said that no matter how good an analytical method may be, one must have at least a fair sample in order to reach a correct result. The preparation of such samples, whereby irregularities in test results could be minimized, was the object the authors endeavored to attain.

Attention may be called to the fact that there is an appreciable difference in the velocity of flow data cited in Passano's discussion (67.5 m. (225 ft.) per min. against 5 m. (16.5 ft.) per min.). The authors have found in their studies on velocity of flow and certain other factors, the results of which have not yet been published, that the mechanism of the corrosion process differs markedly at these two velocities. At the lower velocity (5 ft. per min.) the corroding medium (water) assumes a straight-line flow, while at the higher velocity (225 ft. per min.) the conditions of flow are more turbulent. Thus, the liquid film surrounding the specimens should be affected to a greater extent at the higher rates of flow, and therefore the specimens should be more susceptible to conditions existing in the surrounding body of flowing water. The authors are of the opinion that information gained on the mechanism of corrosion at various velocities of flow would be of value regardless of whether the data lie within or slightly outside of the limits of error as determined by statistical analysis. Nevertheless, there appears to be substantial agreement in the results obtained at relatively low velocities of flow, up to 5 m. (16.5 ft.) per min.

The authors are in agreement with Passano's observations that a complete corroding of the surface is essential when one desires to get a measure of the maximum corrosion of which a specimen of any given material is capable. They realize, however, that difficulty would be experienced in attaining this ideal with various iron or steel objects placed in service in the atmosphere, under water, etc. It has been shown that the presence of an oxide film on the surface of the specimens has a marked influence on the initial corrosion rate of iron at the higher velocities of flow.

THE USE OF BUREAU OF STANDARDS SOIL CORROSION DATA IN THE DESIGN AND PROTECTION OF PIPE LINES[1]

By K. H. Logan[2]

Synopsis

In 1922 the National Bureau of Standards undertook to determine whether serious corrosion could occur underground in the absence of stray electric currents. The existence of such corrosion is demonstrated by the data, but the solution of the problem of economically reducing the corrosion is incomplete. Soils rather than materials control the corrosion of existing pipe lines. The Bureau of Standards data on the corrosiveness of soils must be modified by coefficients or factors which take account of conditions not represented in the Bureau tests. Further work is required to determine the significance of certain tendencies shown by the data; notably, the effects of the size and age of the specimen on which pit measurements are made, the protective effects of corrosion products and the results of departure from homogeneity of the soil with respect to its physical characteristics.

An economic solution of the problem of protecting pipes against corrosion cannot be found until numerical values can be assigned for the life of unprotected pipes and the life extension to be expected as the result of the use of a protective coating. Ideas as to the necessary qualities of a satisfactory coating have been developed but there are no satisfactory ways of measuring these qualities or of determining the amounts of each property which are necessary.

Introduction

By the direction of Congress, the National Bureau of Standards began in 1911 an investigation of stray-current electrolysis and means for its mitigation. In the course of the investigation many instances were found where the condition of the pipe could not be explained by the electrical conditions prevailing at the time. The operators of the street railways claimed that the corrosion was the result of soil action, while the owners of the pipes believed that the observed electrical conditions were not the ones prevailing at the time the corrosion occurred. To settle the question as to the relation of soils to corrosion, specimens of the commonly used ferrous pipe materials were buried, in 1922, in 46 soils, most of which were selected by the U. S. Department of Agriculture as representative of soils containing extensive pipe networks. In most cases nothing was known as to the corrosiveness of the soils.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Chief, Underground Corrosion Section, National Bureau of Standards, Washington, D. C.

The pipes from which the specimens were cut were selected from stock by pipe makers as representative materials. The specimens, 6 in. in length, were filled with earth and buried on end 1 ft. apart at depths of from 2 to 5 ft. depending on the practice of gas and water companies in the region where the test was conducted. Twelve specimens of each material, except cast iron, were buried in each location. Two specimens of each material have been removed from 22 of the more corrosive locations at five 2-year intervals and from 24 less corrosive soils at four 2-year intervals. From time to time specimens of additional materials were buried in the original test locations and ad-

TABLE I.—BUREAU OF STANDARDS SPECIMENS BURIED 1922 TO 1932, INCLUSIVE.

	BURIED	REMOVED	REMAINING IN GROUND, JANUARY 1, 1935
Ferrous	11 070	7 320	3610
Non-ferrous	6 820	4 650	2020
Coatings	3 620	2 560	1025
Miscellaneous	1 130	860	260
Total	22 640	15 390	6915
A.G.A. coatings	2 352	1 176	1176
A.P.I. nipples	2 070	1 380	690
A.P.I. line coatings	2 100	1 260	840
Total	6 522	3 816	2706
Grand total	29 162	19 206	9621

TABLE II.—COOPERATORS IN THE BUREAU OF STANDARDS SOIL CORROSION INVESTIGATION.

	SUPPORTING 1 PROJECT	SUPPORTING 2 PROJECTS	SUPPORTING 3 PROJECTS	SUPPORTING 4 PROJECTS	SUPPORTING 5 PROJECTS
Utilities furnishing sites, labor	56	21	4	1	1
Manufacturers:					
Ferrous	15	6	3	..	..
Non-ferrous	6	5	..	..	..
Coatings (bituminous and metallic)	25	14	5	..	..
Associations	14				
Total number of cooperators	176				

ditional test sites have also been established. The work undertaken by the Bureau of Standards has been supplemented by associations that have stationed research associates at the Bureau to study corrosion problems of special interest to the associations. Table I shows the number of specimens which have been buried and the number which will still be remaining in the ground on January 1, 1935.

The total cost of the investigation from 1922 to January 1, 1935, will approximate $400,000, which is a small fraction of the annual loss caused by underground corrosion. Of the total expended for the study, the share of the Bureau of Standards is roughly 40 per cent. The remainder of the costs have been borne by nearly 200 public utility organizations, pipe and coating manufacturers, and trade associations. This large number of business organizations which have spent earnings to support the various phases of the investigation is probably the best evidence of the need for solutions of underground corrosion problems. Table II indicates the distribution of the cooperation in the investigation.

Some skeptic may ask what return has been received for this large expenditure. A complete answer cannot be given since the saving resulting from the information derived from the investigation depends not solely on the value of the data but on the use which has been made of them. It can be said, however, that the original purpose of the investigation has been accomplished. There is now no doubt that serious corrosion can occur underground in the absence of stray electric currents. This conclusion has done much to modify the attitude of owners of gas and water pipes toward street railway organizations and has undoubtedly led to a saner treatment of underground corrosion problems.

The conclusion that soils may be corrosive immediately raises the question how corrosion by soils can be reduced. This has proved a more important and more difficult question than the one which the Bureau of Standards originally undertook to answer. It is not surprising that the experiments planned to answer the original question should be found inadequate and to some extent unsuitable for solving the newer problem. The question before those interested in underground corrosion is not the adequacy of the form or number of the specimens which have been buried but what use can be made of the data derivable from the buried specimens. The question is pertinent because of the absence of any other large body of data on underground corrosion and of any prospect of adequate data being made available either through additional tests or the assemblage of the experiences of pipe line operators.

The Bureau of Standards data are being extensively used in the selection of pipe and coating materials. It is the purpose of this paper to point out some of the characteristics of the data in order that they may be used more effectively.

SOME CHARACTERISTICS OF THE BUREAU OF STANDARDS SOIL CORROSION DATA

The characteristics of the Bureau of Standards data on soil corrosion which will be commented on in this paper have been discussed in greater detail in the Bureau's publications. The summarizing of the characteristics here is to make more understandable the discussion which is to follow.

Importance of Soil Characteristics.—One of the first observations which were made when specimens were removed in 1924 was that practically all of the specimens from the same soil were corroded similarly. This is illustrated by Fig. 1. From this similarity of corrosion pattern on specimens of different materials, the conclusion has been drawn that the differences in the materials were not sufficient to control the corrosion pattern. This is an important conclusion from the standpoint of the pipe user who has derived his information concerning materials largely from trade literature.

A closely related observation made at about the same time was that in different soils corrosion patterns differed widely. This also is shown in Fig. 1. The two observations suggested that the corrosion pattern is characteristic of the soil to which the specimens are exposed. Additional experiments showed that for the soils investigated, wherever a specimen was exposed to a well-defined soil type as identified by the Department of Agriculture, similar results were obtained. This indicates that corrosion underground is controlled to a large extent by soil conditions which are not accidental but are characteristic of the soil. The importance of soil characteristics is such that, until 1932, the attention of those studying underground corrosion at the Bureau was directed almost exclusively to studies of these characteristics, and the reports which have been published by the Bureau of Standards deal only with the relation of soils to corrosion.

Effect of the Area of the Specimen on the Maximum Pit Depth.—While it is obvious from the data that soil characteristics control the corrosion of the commonly used ferrous materials underground, the expression of this fact in terms of rates of corrosion is not entirely satisfactory. As reported in 1930,[1] specimens of the same material but differing in size differ in the depth of the deepest pits, the deeper pits appearing on the larger specimens. This is illustrated by the data for the 1930 specimens for which the mean of the single deepest pit for all of the $1\frac{1}{2}$-in. specimens is 6.6 ±0.19 mils, while the corresponding figure for the 3-in. specimens is 7.67 ±0.21 mils.

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[1] K. H. Logan and V. A. Grodsky, "Soil Corrosion Studies, 1930. Rates of Corrosion and Pitting of Bare Ferrous Specimens," National Bureau of Standards *Journal of Research*, Vol. 7, July, 1931, p. 1.

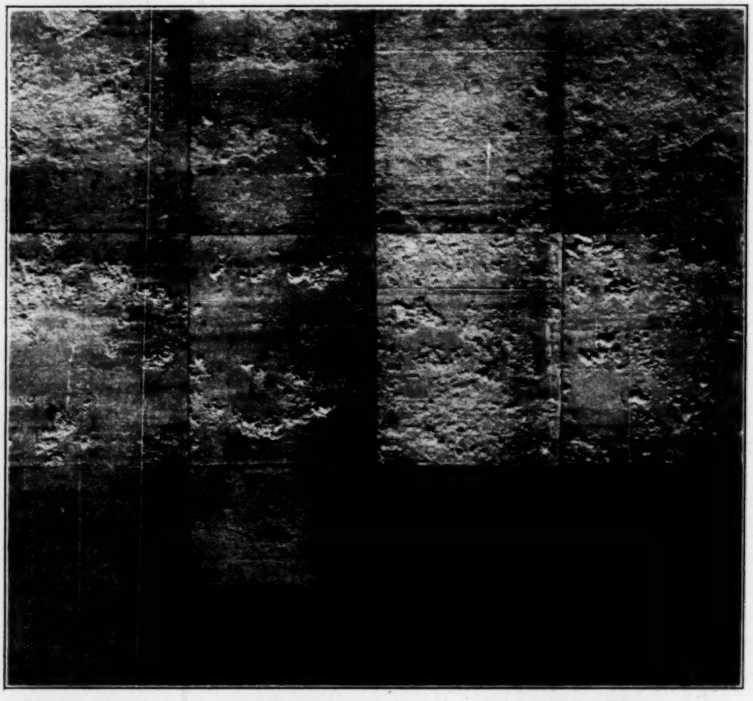

FIG. 1.—Panoramic Pictures of 5-in. Lengths of $1\frac{1}{2}$-in. Pipes.

Note similarity of corrosion patterns. Top row, two specimens of pure open-hearth iron and one each of wrought iron and Bessemer steel exposed 10 years to Hempstead silt loam; second row, same materials exposed 10 years to Ontario loam; bottom row, same material exposed 8 years to a wet silt loam in Salt Lake City.

Scott[1] expressed the relation between the maximum pit on specimens of different sizes exposed to the same conditions as:

$$\frac{P_2}{P_1} = \left(\frac{A_2}{A_1}\right)^a \qquad (1)$$

where P_1 and P_2 are maximum pit depths on areas A_1 and A_2 respectively and a is a constant which is a function of the conditions of exposure. The application of the formula to Bureau of Standards specimens would involve the computation of values of a for each soil for each period of exposure. The formula has been applied to a few of the Bureau data in adjusting the depths of pits for size of specimen, but it has not yet been possible to determine whether this method would yield more accurate results than the arbitrary adjustment used in reporting the soil corrosion data in the Bureau of Standards *Journal of Research.*

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[1] G. N. Scott, "Report of A. P. I. Research Associate to the Committee on Corrosion of Pipe Lines," *Proceedings*, Am. Petroleum Inst., Vol. 14, No. IV, November, 1933, p. 204.

This latter method consists in using the single deepest pit on each of the 1½-in. specimens, the two deepest pits on each of the 3-in. specimens, and the four deepest pits on each of the 6-in. specimens. The exposed area of a 1½-in. specimen is roughly 0.23 sq. ft. The pitting rates in the reports referred to are based on the average maximum pit as defined above on two specimens of each of the rolled materials and on one or two specimens for the cast iron. Thus, the determination of the rate of pitting on the Bureau specimens is equivalent to the measurement of one pit on each 0.1 linear foot of 8-in. pipe or the averaging of ten pit depths per foot of pipe. Obviously the resulting rate of penetration is less than that to be obtained by taking the single deepest pit on, say, a 4-ft. length of pipe exposed for examination or on a 20-ft. length of pipe.

It is interesting to estimate the pit depth to be expected on a 20-ft. length of 8-in. pipe, the area of which is 45 sq. ft., by applying Scott's formula to Bureau data. Treating the data as if soil characteristics did not affect the value of a, Scott found the average value for a for the five periods for which data are available to be approximately 0.16. For seven pipe lines on which he secured pitting data his average value for a is 0.26. If the lower value of a is adopted, the Bureau data should be multiplied by the factor 2.36 or, if the larger value of a is chosen, by 4.0 in order to obtain the average maximum pit on a 20-ft. length of 8-in. pipe. The reader is not to understand that either of the factors is recommended for applying Bureau data to pipe lines. They are based on averages and the assumption that all of the soils from which the data were derived are similar, whereas it has been shown that the Bureau tests cover a number of soils which differ in many of their fundamental characteristics. The values for a for the individual soil types have not been determined, and until this has been done, the Bureau data cannot be reduced to field conditions by means of Scott's formula. Moreover, there may be corrections other than for size of specimens that should be applied to the Bureau data.

It will be noted that the exponent a which Scott found for the

Bureau data is much smaller than that for his field data. A possible explanation for this is that trench conditions for the Bureau specimens were more nearly uniform than usually obtain in the field. Moreover, pipe lines are affected by additional conditions affecting corrosion, such as those causing long-line currents. Because it is very difficult to secure satisfactory field corrosion data, it is very important that a coefficient be found which will permit the direct application of the Bureau data to pipe-line construction.

Dispersion of Bureau Data.—An interesting and disconcerting fact concerning the Bureau data is that two specimens of the same material, buried for the same time in the same trench, may differ widely in the depth of the deepest pits. This is illustrated in Table III. The dispersion of the data for the specimens in the tidal marsh is typical of the less uniform sets of data, and that for Susquehanna clay of the more uniform sets. The uniformity of corrosion in some soils is illustrated by the specimens in the first two vertical columns in Fig. 1. Sometimes there is evidence that one part of the trench is more corrosive than another, but often there is no apparent explanation for the differences in the specimens.

TABLE III.—MAXIMUM PIT DEPTHS OF BUREAU OF STANDARDS SPECIMENS IN TWO SOILS.

Material	Identification Letter	Maximum Pit Depth, mils		Difference in Pit Depth, mils	Distance Between Specimens, in.	Average Deepest Pit, mils	
		No. 1	No. 2			Unweighted	Weighted
SPECIMENS IN TIDAL MARSH (SOIL NO. 43)							
Wrought iron	b-d	102.0	50.0	52.0	138	76.0	76.0
Bessemer steel	M-M	149.5	82.0	67.5	107	115.7	101.9
Bessemer steel	e-e	77.5	63.5	14.0	79	70.5	70.5
Open-hearth steel + copper	Y-Y	94.0	216.0	132.0	67	155.0	135.7
Wrought iron	B-D	148.0	123.5	24.5	50	135.6	129.0
Open-hearth iron	a-a	181.0	44.5	136.5	40	112.8	112.8
Bessemer steel	y-y	81.5	64.5	17.0	25	73.0	73.0
Open-hearth steel	K-K	112.5	86.5	36.0	18	99.5	94.6
SPECIMENS IN SUSQUEHANNA CLAY (SOIL NO. 42)							
Wrought iron	b-d	80.0	88.5	8.5	138	84.3	84.3
Bessemer steel	M-M	108.0	98.5	9.5	107	104.0	98.4
Bessemer steel	e-e	92.5	115.5	23.0	79	104.0	104.0
Open-hearth steel + copper	Y-Y	111.5	96.0	15.5	67	103.7	99.4
Wrought iron	B-D	106.0	86.0	20.0	50	96.0	94.0
Open-hearth iron	a-a	79.0	88.0	9.0	40	83.5	83.5
Bessemer steel	y-y	86.0	98.5	12.5	25	92.3	92.3
Open-hearth steel	K-K	133.0	125.0	8.0	18	129.0	121.5

Two conclusions can safely be drawn from the dispersion of the data. Since it has been shown that the various kinds of ferrous materials behave similarly and since the sites for the test were selected by soil experts and the trenches filled by men familiar with the laying of pipe lines, it is evident that the rate of corrosion is markedly affected by differences in trench conditions too small to be noticed by ordinary observers. That these accidental trench conditions have more than temporary effects is indicated by the lack of uniformity in the 10-year corrosion data. It follows from the above conclusion that accidental conditions so greatly modify rates of underground corrosion that the relative merits of materials cannot be determined by data derived from a few exposures of different materials to approximately similar conditions. This is illustrated in Fig. 2, which shows the conditions of the 1½-in. wrought-iron and bessemer steel specimens from the tidal marsh for which data are given in Table III. The wrought-

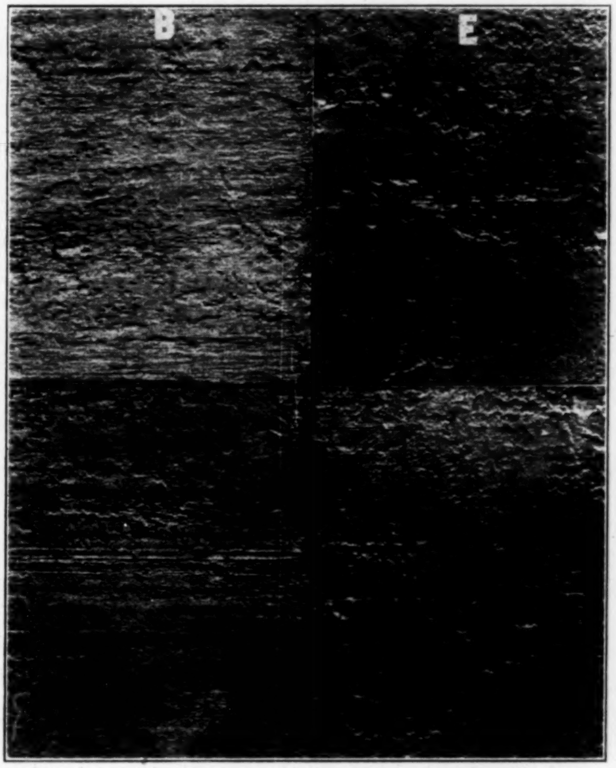

FIG. 2.—Wrought Iron, on Left, and Bessemer Steel, on Right, Exposed 10 Years in a Tidal Marsh.

Note difference between two specimens of the same material.

iron specimen on one side of the trench was pitted more deeply than the bessemer steel specimen on the same side of the trench, while on the other side of the trench the reverse is true. The table also indicates that one specimen of open-hearth iron pitted more deeply than any other specimen except one in either of the two soils for which data are given, while the other representative of this material in the same soil showed the least pit depth. Thus, even when care is taken to secure uniform test conditions, accidental conditions may cause two specimens of the same material to yield quite different data.

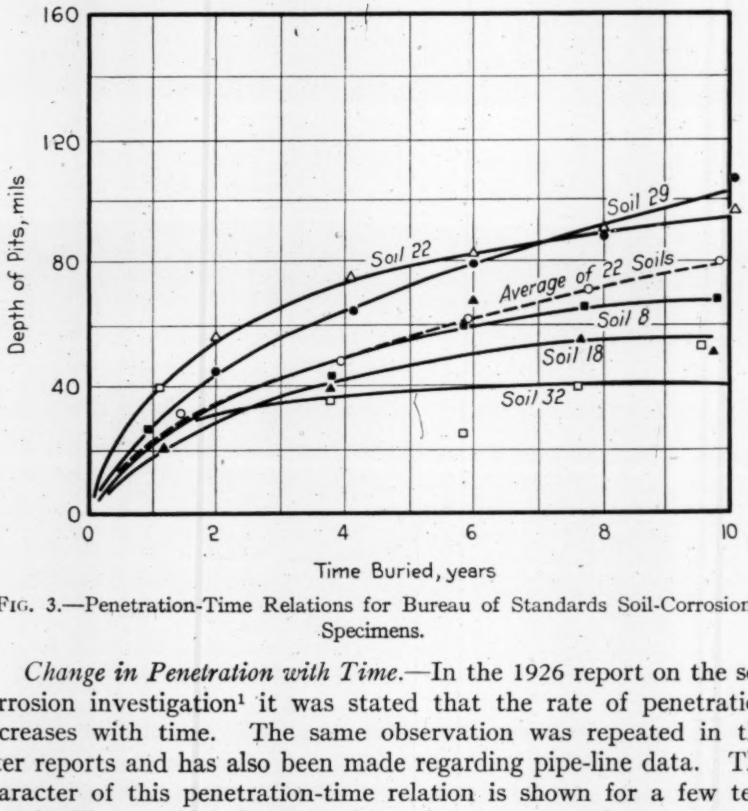

FIG. 3.—Penetration-Time Relations for Bureau of Standards Soil-Corrosion Specimens.

Change in Penetration with Time.—In the 1926 report on the soil corrosion investigation[1] it was stated that the rate of penetration decreases with time. The same observation was repeated in the later reports and has also been made regarding pipe-line data. The character of this penetration-time relation is shown for a few test sites in Fig. 3 which is taken from the report on the 10-year-old specimens.[2] Scott expressed the relation between pit depth and time by a hyperbolic equation, stating that one of the constants in this equation was nearly the same for all soils while the other was a function of the soil. On this basis the ultimate depth of penetration in any soil can be computed from a single set of observations of pit depths after a single period of exposure; that is, by determining the maximum penetration at a representative number of points on a pipe line of a known age, the ultimate depth of the corrosion can be estimated. Obviously any such estimation must take account of the area of the pipe surface as indicated in Eq. 1 and the precision of the measurements of pit depths as indicated by the standard error of the data. Since any mathematical process is but a form of logic, the reliability of the results of the process depend on the validity of the assumptions upon which the computations are based, as well as on the validity of the process itself. The user of the formulas referred to must therefore consider not only the standard error of his results but the validity of the fundamental assumptions from which the formulas were derived. Scott's equation for the penetration-time relation was presented as an empirical formula justified by observation of pitting data and supported by theoretical considerations.

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[1] K. H. Logan, S. P. Ewing, and C. Yeomans, "Soil Corrosion Studies I," National Bureau of Standards *Technologic Paper No. 368*, April, 1928.

[2] K. H. Logan and R. H. Taylor, "Soil Corrosion Studies, 1932," National Bureau of Standards *Journal of Research*, Vol. 12, January, 1934, p. 119.

It has been shown above that buried iron is very sensitive to trench conditions. Everyone experienced in the uncovering of buried pipes realizes that trench conditions change radically with time. When the trench is newly backfilled, unless it is puddled or carefully tamped, the backfill is lumpy and full of voids which allow both air and water to reach the pipe readily. The supply of oxygen varies greatly from point to point and this has been shown by Aston, Evans and others to be favorable to corrosion. As the backfill settles, conditions become more uniform and therefore less favorable for corrosion; consequently, the curvature of the pitting-time curves may be the result of changes in trench conditions rather than the accumulation of corrosion products. This would not affect the validity of the equation for these curves, but it has an important bearing on the relation of pipe coatings to corrosion, which will be discussed later.

THE APPLICATION OF UNDERGROUND CORROSION DATA TO FIELD PROBLEMS

Fundamental versus Practical Data.—While the data on underground corrosion may be of interest as a source of information concerning the fundamental processes which result in the deterioration of pipe underground, and while the ideal way to prevent corrosion is to base mitigative measures on fundamental principles, the great majority of inquiries concerning underground corrosion come from manufacturers and consumers who wish specific information by means of which they can obtain more economical transportation of their fluids. For these men, statements of fundamental principles, average rates of corrosion, and average performance of materials are of little interest because the statements cannot be directly and immediately applied to the construction problems which the inquirers have in mind. Many of these men seem to prefer a material which will insure satisfactory service to one which can be shown by statistical analysis to be, on the average, more economical, just as many have a tendency to purchase the higher priced of two similar articles because they believe that somehow it must be better, and they desire the best. This is not wholly illogical since in any mathematical analysis we are forced, for purposes of simplification, to ignore apparently unimportant factors which, under some circumstances, may become important. Thus, in estimating the most economical pipe line, it is usual to ignore the costs which may result from interruption of service and the damages caused by leaks and explosions because there is no satisfactory way of evaluating these factors in advance. To provide against these contingencies, the engineer uses factors of safety based on experience and intuition. In the laying of pipe lines such factors appear in the form of more expensive materials, thicker pipe walls, or protective coatings.

Determination of the Need for Protection.—At the third Bureau of Standards Conference on Underground Corrosion the members of the Underground Corrosion Section presented a paper entitled "A Suggested Procedure for Determining the Protective Needs of Pipe Lines Against Soil Corrosion." This paper has not been published because the authors, while they agreed upon the principles to be followed, were unwilling to assign specific values to the constants in their formulas and consequently left the formulas in an unusable condition. Subsequent to the conference some progress has been made in determining the ultimate depth of pits. There remains the assignment of values to protective coatings, but pipe line protection has taken on a somewhat different aspect. This will be discussed later.

The Bureau of Standards corrosion data indicate that while a small percentage of the soils of the United States are quite corrosive, the majority of the soils are only mildly corrosive or non-corrosive. Among the first things to be considered in the laying of a pipe line is, therefore, the question of whether or not corrosive soil will be encountered, since it would not be economical to protect the entire length of a line if only a small part of it is to be exposed to corrosive soils.

The more commonly used ways of determining the corrosiveness of soils can be briefly discussed. Measurements of soil resistivity have been satisfactory where alkali soils are responsible for corrosion, but no general correlation between soil resistivity and corrosion has been found. Shepard[1] modified and extended the use of soil resistivity as an indicator of corrosiveness by pointing out the fact that in the south central portion of the country a corrosive soil was one that was much lower in resistance than the surrounding soils but was not necessarily a low-resistance soil. He did not offer an explanation for this correlation although in the light of the theory of differential aeration one can be given. The relatively low-resistance soils usually contain more moisture, hence less oxygen reaches the pipe in such soils. By reason of the exclusion of oxygen the pipes in the more moist soils are anodic.

Denison[2] correlated the total acidity of soils at the Bureau test sites with the depth of pits on the test specimens and indicated a correlation between soil acidity and the corrosion of a group of pipe lines in a level section of Ohio. He and Grodsky also developed a laboratory test using steel disks which was satisfactory for acid soils on the basis of the Bureau data. However, when Ewing[3] attempted a correlation between replacements and soil acidity for a group of pipe lines extending over several hundred miles, the correlation was unsatisfactory. It is possible that the corrosion which can be associated with soil acidity was modified by that associated with the potentials set up between soils differing in their permeability to air. Ewing observed that each soil type had roughly the same corrosiveness wherever he encountered it. This was to be expected since all soils belonging to the same soil type are similar in their chemical and physical characteristics. It follows that if the corrosivity of all soil types were known the anticipated corrosion of a pipe line could be determined by identifying the soil types traversed by the line.

When the correlation of the Bureau tests with pipe-line corrosion has been completed the corrosivity of about 75 soil types will be known. The corrosiveness of other soil types can be obtained by determining the types of soils for which the gas and oil pipe line operators have corrosion data. Still other correlations can be based on the similarity of different soil types.

The proper method of expressing the corrosiveness of a soil has not been generally agreed upon. There is reason to believe that during the first few years of exposure the corrosion is not proportional to the period of exposure and must be expressed by an equation such as that suggested by Scott. Where soils are not very corrosive or where the thickness of the pipe wall insures a long pipe life, the effect of the first few years during which corrosion is most rapid may be neglected and a straight-line depreciation curve may give as satisfactory results as a curve expressed by a more complicated equation, since in any event the result can represent only an average value having a large standard deviation.

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[1] E. R. Shepard, "Pipe Line Currents and Soil Resistivity as Indicators of Local Corrosion Areas," National Bureau of Standards *Journal of Research*, Vol. 6, April, 1931, p. 683.

[2] I. A. Denison, and R. B. Hobbs, "The Corrosion of Ferrous Metals in Acid Soils," to appear in the National Bureau of Standards *Journal of Research*, Vol. 12 (1934).

I. A. Denison, "Correlation of Certain Soil Characteristics with Pipe Line Corrosion," National Bureau of Standards *Journal of Research*, Vol. 7, October, 1931, p. 631.

[3] S. P. Ewing, "Corrosion Surveys for Pipe Lines and Distribution Systems," to be published in the Am. Gas Assn. Monthly.

If rates of penetration of pipe lines were no greater than those of Bureau of Standards test specimens, there would be few localities in which corrosion would be a problem when any except thin-walled pipes are used, and corrosion problems would be confined largely to small-diameter pipes, such as house services. Since the cost of the pipe in such cases is frequently half or less of the total cost of the installation, the problem can be economically solved by the use of a thick-walled pipe or one of a non-ferrous metal. It appears, however, that the corrosion of pipe lines is a more complicated matter than the corrosion of short lengths of pipe, probably because of the introduction of differential aeration resulting from the passage of the line through soils differing in texture or other property, whereby additional galvanic currents are set up. For example, the experience of a pipe-line operator who has a line near one of the Bureau test sites indicates that his pipe corrodes many times faster than the Bureau specimens. An operator in California has had a similar experience. On the other hand, there are places where the Bureau data indicate more rapid deterioration than pipe line experience shows for the same soil. An explanation is that in one case the direction of the galvanic currents is such as to accelerate corrosion in the soil in which the Bureau specimens were tested, while in the other case they afford cathodic protection. However, it is reasonable to expect that the direction of the flow of current can be predetermined by a study of topography and of soil types and textures. It has already been explained that the larger the exposed area the deeper will be the deepest pit. Consequently the Bureau data should be adjusted by a factor which depends on the ratio of the area of the Bureau test unit to the area of the pipe line under consideration.

The Prevention of Corrosion.—If experience or tests indicate that a soil may be corrosive, the first thought of the inexperienced is the use of a corrosion-resistant material. The Bureau tests indicate that at least for very corrosive soils this would involve non-ferrous materials or ferrous alloys at least equally expensive. Perhaps in the future plymetal may afford a solution. The other commonly considered method for reducing corrosion losses is the use of protective coatings. To select proper coatings we must discover the cause of the shape of the penetration-time curves such as are shown in Fig. 3. If, as has been suggested, the decrease in the rate of penetration with time is largely the result of the protective effects of corrosion products, then the life extension of a pipe line resulting from the application of a coating is roughly equal only to the life of the coating. If, however, the bending in the pitting-time curves is caused largely by the reduction of potential differences resulting from more uniform trench conditions produced by the settling of the backfill of the trench, then the life extension resulting from a coating is much greater. On the latter assumption, the coating in protecting the pipe while the trench settles, acts as if it transferred the pipe from a corrosive to a non-corrosive soil. Under one theory the function of a coating is to prevent corrosive soil solutions from reaching the pipe. Under the other theory the function of the coating is to reduce the differences in the concentration of oxygen at the pipe surface until a more uniform condition is established by the settling of the soil.

One theory calls for an impervious coating, the other for a coating of uniform texture sufficiently impervious to control the supply of oxygen. It is obvious that the objective of the A.P.I. coating tests, stated by Bly[1] as "to inject engineering principles into the design and selection of protective coatings for underground pipe lines" cannot be accomplished until the merits of these two theories have been determined. It should be evident also that it is impossible to decide whether a pipe line should be protected or not without making some explicit or implicit assumption as to the life of an unprotected pipe and the life extension resulting from the use of a protective coating. It is not sufficient to decide whether or not the soil is corrosive or that one coating is better than another. It is important, therefore, that makers and users of pipe fully understand the present status of data on pipe-line corrosion and protection, and that they appreciate what remains to be done before the data now available can be used intelligently.

The Bureau tests indicate that pipes well coated with lead or zinc begin to show rust spots in corrosive soils in from six to eight years. As has just been indicated, it is impossible to state at this time whether this period is the life extension of the pipe resulting from the use of the coating.

The dispersion of the data and the limited number of specimens make it impossible to determine whether one base material is better than another for the production of galvanized pipe. There is some indication that a heavy coating of zinc is superior to a light one on a metal pipe. While the rate of deterioration of metallic coatings seems to differ widely with different soils, it cannot be said that some metallic coatings are particularly suitable for certain soil types. Thus, galvanized sheet steel resisted corrosion very well when exposed to Susquehanna clay—a heavy, acid soil—but showed pitting in several less acid soils as well as in the alkali soils after eight years' exposure. These indications are confirmed by the behavior of the specimens removed after shorter periods of exposure.

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[1] P. Bly, Progress report of the pipe line and production subcommittee of the general committee on corrosion, *Proceedings*, Am. Petroleum Inst., Vol. XI, p. 113 (1929).

The most commonly used pipe coatings are bituminous, and it is in connection with these coatings that there is the greatest need for scientific data. While, as a result of the work of Scott[1] and Ewing,[2] it is now generally recognized that a bituminous coating should resist soil stress, should adhere to the pipe, should not shatter under shock, and should not flow when warm or crack when cold; there are no data which will support the specification of definite numerical values for these properties. It is thought that a bituminous coating should be moisture-proof; but however low the conductivity is when the coating is applied, it almost invariably shows a large increase in conductivity after a year's exposure to moist soil. A coating conductance of less than 10 micro-mhos per sq. ft. has nearly always indicated perfect protection.[1]

There is a great need for the data which will permit the specification of the limiting values of the necessary qualities or properties of bituminous coatings and for recognized methods for testing both coating materials and finished coatings. Before this need can be met, someone must determine how long a pipe line needs protection.

CONCLUSION

The work done on underground corrosion and its mitigation, by members of the Bureau of Standards staff and by those cooperating with the Bureau has brought to light many new and useful facts concerning the deterioration of pipe lines.

At present the data are inadequate for the solution of the corrosion problem but they constitute a basis for, and point the way toward, the solution. The greatest immediate needs with respect to underground corrosion are more general knowledge of the character and significance of the data which have been obtained and a clearer realization of the data which can and must be secured in order to complete the solution of the underground corrosion problem.

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[1] G. N. Scott, "A.P.I. Pipe Coating Tests," *Proceedings*, Am. Petroleum Inst., Vol. 12, No. IV, p. 55 (1931).

[2] S. P. Ewing, "Pipe Coatings and Corrosion", *Proceedings*, Am. Gas Assn. (1933).

DISCUSSION

MR. F. N. SPELLER[1] (*presented in written form*).—I should like to point out one or two characteristics of these data and compare them with the results of other investigations.

Looking back over the work of the Society's Committee A-5 on Corrosion of Iron and Steel and the 10-year soil corrosion tests, it now seems clear that there is no material difference in corrosion resistance between the various types of ferrous metals commonly used, when exposed to water or soil. This conclusion was predicted from shorter tests by the writer and others 20 years ago and has been amply verified by long-time tests such as those conducted under Mr. Logan's direction. The accompanying Fig. 1 summarizes the weighted average penetration in each of these materials exposed in 22 of the more corrosive soils for 10 years. Local electrochemical action is much more pronounced in soil corrosion so that naturally more variation between individual samples in the same type of soil is found.

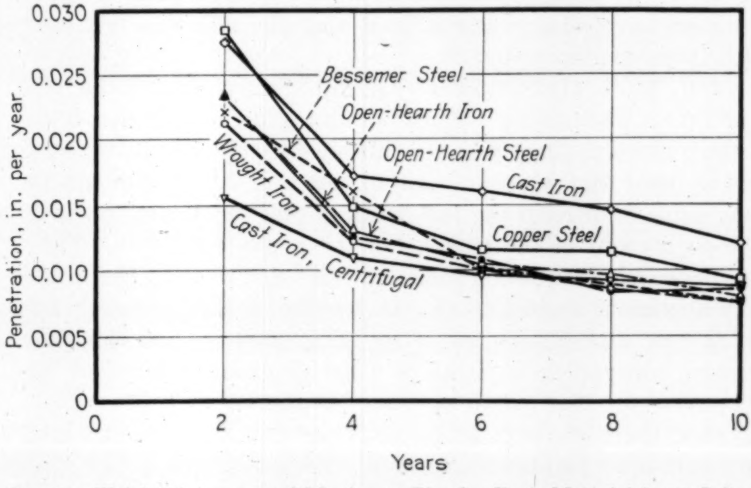

FIG. 1.—Weighted Average of Maximum Pits for Each Material in 22 Soils.

Perhaps the loss due to maintenance and replacement of pipe underground has been exaggerated. As a matter of fact, most soils offer no problem in corrosion. On the whole, less than 10 per cent of the 400,000 miles of pipe now in use underground requires protection within the period of life for which it was designed. A few pits penetrating one-half of the wall thickness do not reduce the strength of the pipe. Many lines are laid with little or no protection, as it has been found more economical to replace short stretches in "hot spots" than to put costly coatings on the whole line. Moreover, when leaks occur, they can now be readily welded and the pipe placed in as-good-as-new condition at a fraction of the cost of new materials.

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[1] Director, Department of Metallurgy and Research, National Tube Co., Pittsburgh, Pa.

Mr. Logan has pointed out the greater need for data which will permit specifications for underground pipe coatings and practical methods for testing such coatings. Subcommittee XXVI on Underground Pipe Protection of the Society's Committee D-1 on Preservative Coatings for Structural Materials has taken the first step toward this in formulating specifications for bituminous primers and will endeavor to develop methods of testing the adhesive properties of bituminous coating materials.

MR. J. T. MACKENZIE.[1]—Mr. Speller has shown the relative penetration rates of the different products in the corrosion tests and as these items appear, they are entirely correct. However, we must bear in mind that when he came to cast iron, Mr. Logan was put to some pains to develop the real depth of pits which were made in the pipe on account of the tenacity of the graphite residue. The Cast Iron Research Association some six years ago went to the trouble to bury specimens consisting of 2-ft. lengths of 6-in. cast-iron pipe to which were attached means of applying pressure, and this year the pressure was applied to some of these specimens which had been in the ground for that length of time. In the soil at Buttonwillow, for instance, there was a puncture in one of the Bureau's cast-iron test specimens after two years' exposure. The Association's 2-ft. specimen of cast iron withstood 450 lb. internal pressure without any loss of pressure during the test. Also, at Niland and at New Orleans, probably the three worst soils in the whole test, the bombs were tested without failure. The graphitic residue left on cast-iron pipe, I think, has probably been underestimated, and is probably an explanation of the long life of cast-iron pipe in service. There are a great many users of cast-iron pipe who will tell you that they have no trouble from corrosion, whereas, as a matter of fact, if you dig up the lines and take a hammer you can go through the pipe in many places. According to Mr. Logan's theory, the action of clods is probably responsible for this, because we have a rather poor coating on the pipe, such as is common with tar coatings usually applied, and may expect localized pittings instead of general action. Cast iron, as usually supplied, is of much greater thickness than the wrought material, so a considerable area may be corroded but will continue for a long time to carry the pressure, whereas a steel pipe would have failed long before. I think we can reconcile most of the facts of experience with the facts developed in this corrosion test of the Bureau of Standards.

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[1] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

MR. K. H. LOGAN.[1]—Mr. MacKenzie's experience is borne out in a condition I discovered in a town in New York. There are two lines of pipe of different materials, and it happens that the older and thinner walled line has given very much less leakage trouble than the newer line. The man who has charge of these lines tells me that he is afraid to disturb this older line, because he thinks that if he disturbs it, it will go to pieces, that he has an incrustation over that line which is holding it together. So it may be that corrosion products are of value not only in cast iron but in other kinds of iron as well.

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[1] Chief, Underground Corrosion Section, National Bureau of Standards, Washington, D. C.

A HIGH-SPEED FATIGUE TESTING MACHINE AND SOME TESTS OF SPEED EFFECT ON ENDURANCE LIMIT

By G. N. Krouse[1]

Synopsis

The author presents a shorter and less-expensive method of determining the endurance limits of materials by the use of a high-speed fatigue testing machine driven by an air turbine, operating over a range of speeds from 5000 to 30,000 r.p.m. Two inexpensive specimens are described with the critical section approaching the ideal and worst surface conditions.

The results of tests on four steels, two cast irons, brass and duralumin, covering a wide range of endurance limits and speeds are presented. Check tests made on the slower-speed rotating beam machine show that the speed effect may be estimated with a fair degree of accuracy and is of small import when compared with the effect of surface finish.

A major difficulty in the general adoption of repeated-stress tests of metals is the long time required to determine the endurance limits. Using a speed of 1500 r.p.m. it takes about a week to determine an endurance limit for a ferrous metal, and much longer for some non-ferrous metals. Various accelerated tests have been proposed, but none as yet devised have proved thoroughly reliable. Increasing the speed of cycle applications seems to be a more promising method of shortening the time required to determine the endurance limit, provided the speed effect may be estimated with a fair degree of accuracy. The rotating-cantilever-beam type of fatigue machine seems well fitted for high-speed operation and a directly connected air turbine a most satisfactory driving motor.

Figure 1 shows the completed machine which operates under air pressures ranging from 10 to 40 lb. per sq. in. This machine has been in successful use for over a year in the Materials Testing Laboratory of the University of Illinois.

The specimen, *S*, is gripped at one end by the driving chuck *C*, tightened through an opening in the machine casing above the chuck by means of a pair of end wrenches. The other end of the specimen is held in a similar chuck *C′*, on one end of the loading arm *A*, the other end of which rotates in the loading bearing *B*. The shot container *W*, is supported on the bearing *B* by a slender rod guided through holes in the support *E*. The ball-bearing turbine shaft is driven by a disk rotor *R*, which, in turn, is driven by a high velocity blast of air impinging on the rotor blades. Air under pressure is fed to nozzles through a quarter-turn valve *V*. When a specimen fails, the bearing *B* and the shot container *W* drop on the cut-off button *O*, causing the spring-actuated air valve *V* to close, stopping the turbine. A revolution counter *H* driven by a 100 to 1 worm gear reduction, records the number of cycles of stress.

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[1] Special Research Assistant, Materials Testing Laboratory, University of Illinois, Urbana, Ill.

Continuous, constant-speed operation of the machine, and satisfactory wear of the bearings depends largely upon proper lubrication which is provided by a continuous spray of oil. In Fig. 1 the air-tight oil tank *T* is maintained under air-line pressure through tube *Q*. Tube *F* leads from tube *Q* to the low-pressure side of the turbine where it ends in a small jet *J*, directed into the ball race near the rotor *R*. A regulated amount of oil, flowing from the tank *T* through the needle valve *N* into the tube *F*, is caught in the air stream and issues from the jet *J* in the form of a fine spray. Since the differential pressure across the needle valve *N* is due principally to the static head of oil above the valve, changes in air-line pressure do not affect the oil flow. The oil spray, after emerging from the bearing near the rotor *R*, is caught up by the exhaust air and carried along the turbine shaft through the ball race and exhaust ports to the atmosphere. The exhaust air and oil spray are deflected downward by the surrounding machine casing and the oil is caught in the bottom of a galvanized iron box, which incloses the entire machine while in operation. The collected oil, after filtering through several layers of cloth, may be used again. Less than a half gallon of oil has been used on the machine to date. One tank full of oil, about a third of a pint, will keep the machine properly lubricated for four days when running at 10,000 r.p.m. and one day when operating at full speed.

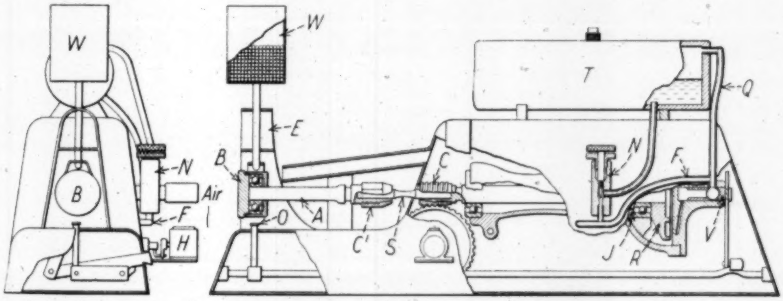

Fig. 1.—High-Speed Fatigue Testing Machine.

It has been found, as will be shown, that within the speed range of this machine, 5000 to 30,000 r.p.m., even a considerable variation of speed has little effect upon the test results, hence the machine speed may be controlled satisfactorily by means of a gate valve on the air supply line. A pressure gage between the gate valve and the machine shows the pressure available, 10 lb. per sq. in. being sufficient to operate the machine at 10,000 r.p.m. while 40 lb. per sq. in. will operate the turbine at 30,000 r.p.m.

The loading weight on the specimen consists of the combined weights of the bearing *B*, shot container *W*, lead shot and a proportionate part of the loading arm *A*. The lever arm of the loading weight is the distance between the axis of the rod supporting the shot container and the critical section of the specimen. This distance is measured by means of a trammel divider, one leg of the divider being placed in the holes of the weight support *E* and the other leg adjusted to reach the critical section of the specimen. The distance between the divider points is measured along a scale. The maximum stress in the specimen is computed by the ordinary flexure formula.

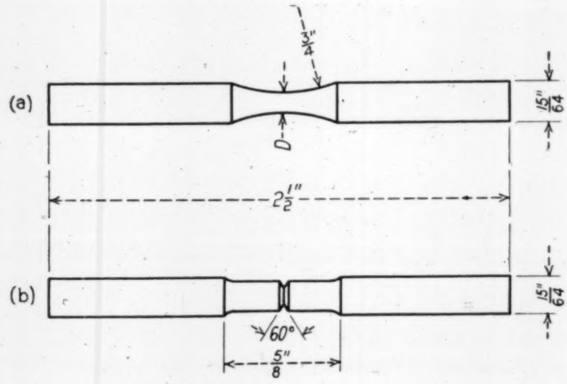

FIG 2.—High-Speed Test Specimens.

(*a*) Polished specimen. (*b*) V-notch specimen.

The endurance limit of a material, with a surface finish approaching the ideal condition, is seldom a true measure of the maximum usable stress under actual service conditions. The stress concentrations due to tool marks, holes and sharp notches are especially pronounced in some metals and should be considered in the selection of a material where repeated stress is the governing factor. With these facts in mind, two specimens have been designed to bring out these important characteristics of a material.

SPECIMENS

Figure 2 (*a*) shows the high-speed polished specimen. It is shown in Table I that the $\frac{3}{4}$-in. radius used on the high-speed specimen does not affect the endurance limit of any material when compared with the 5-in. radius used in the R. R. Moore and Illinois machines. The high-speed specimen is easily given the more desirable axial polish by rotating slowly in a lathe and applying a polishing arbor, rotating about 1800 r.p.m., with its axis at right angles to the axis of the specimen. The diameter D varies between 0.1 and 0.2 in. depending upon the strength of the material.

TABLE I.—ENDURANCE LIMITS OF SPECIMENS TESTED AT VARIOUS SPEEDS.

Material	Endurance Limit, lb. per sq. in.					Endurance Limit Ratios	
	Illinois Machine	High-speed Cantilever Machine					
	1500 r.p.m.	10,000 r.p.m.	30,000 r.p.m.	10,000 r.p.m.	30,000 r.p.m.	10,000 r.p.m.	30,000 r.p.m.
	Polished Specimen			V-Notch Specimen			
S.A.E. No. 1020 steel	31 000	31 000	33 000	22 000	25 000	1.41	1.32
Stainless steel	60 000[a]	63 000	69 000	26 000	28 000	2.42	2.47
S.A.E. No. 4140 steel	98 000 79 000[a]	99 000	102 000	46 000	47 000	2.15	2.21
Rail steel	50 000	50 000	51 000	26 000	27 000	1.92	1.89
Gray cast iron	10 000	10 000	11 000	13 000[c]	13 000	0.77	0.85
Alloy cast iron	26 000	26 000	29 000	22 000	24 000	1.18	1.20
Brass[b]	20 000	24 000	27 000	12 000	13 000	2.00	2.08
Duralumin[b]	15 000	15 000	17 000	12 000	13 000	1.25	1.31

[a] Transverse polish. It has been found on several occasions that certain materials have their endurance limits markedly decreased by a transverse polish.

[b] Endurance limits on basis of 50,000,000 cycles.

[c] The increased endurance limit for the notched gray cast iron has been carefully rechecked.

Figure 2 (*b*) is the drawing of the V-notch specimen adopted to show the susceptibility of the material to a surface finish approaching the worst condition, neglecting corrosion and high-temperature fatigue. Tests by the author prior to the adoption of this V-notch specimen have shown little effect of variation of the ratio of the root diameter to the depth of notch on the endurance limit within the loading range of the machine. The V-notch used in these tests was about 0.01 in. deep with a radius at the bottom of about 0.005 in., as determined at a magnification of 150.

REPRESENTATIVE TEST DATA AND CONCLUSIONS

An attempt was made to select materials covering a wide range of endurance limits. Table I gives the results of five tests using different speeds and finishes on each of eight materials.

From the results of these tests it is shown that up to 10,000 r.p.m. there is no appreciable speed effect when compared with the slower-speed rotating-beam tests. At 30,000 r.p.m. there is an increase in endurance limit varying from 1000 to 6000 lb. per sq. in. for the axially polished specimen while the endurance limits for the V-notch specimens increased up to 3000 lb. per sq. in. In each case the stress concentration factor for the V-notch in a given material remained practically constant. Within practical limits the speed effect may be estimated.

The economy of higher-speed repeated stress testing using the less expensive specimens warrants thoughtful consideration.

DISCUSSION

MESSRS. O. J. HORGER[1] and J. L. MAULBETSCH[1] (*presented in written form*).—The speed effect with polished and notched specimens submitted in Table I of the paper is extremely interesting. The endurance limit ratios for gray cast iron, while indicated as being carefully rechecked by the author, do not seem logical, but no explanation is given for the fact that the ratios are less than 1.

It would appear that the nominal stress at the root of the V-notch specimen was used to determine the endurance limit of those specimens. In the case of cast iron there are many test data indicating that the surface condition of the specimen is of small importance.[2] From these particular tests where the notch is only 0.01 in. deep, it seems logical then to assume that such a notch has about the same effect as the inherent inhomogeneities of the material.

Otherwise it would seem to indicate, as in Table I, that cast iron is strengthened by a circumferential notch. The writers believe that V-notch specimens with so small a diameter as the ones used by the author are not representative for surface condition study. In the case of an 0.2-in. diameter, the groove 0.01 in. deep reduces the section modulus by 27 per cent, which means that not only the surface conditions are varied, but that also the nominal stress is varied by a very large amount. The question is raised then as to how much reduction is due to the change of section modulus and how much due to surface conditions.

MR. J. B. KOMMERS[3] (*presented in written form*).—One of the results in Table I of the paper, in which I was particularly interested, was that on gray cast iron, which showed a higher endurance limit for the notched specimen than for the smooth specimen. A number of years ago I made some tests on a low-strength gray cast iron with a square notch in which the endurance limit was slightly higher for the square-notched specimen than for the smooth specimen. I should like to ask Mr. Krouse whether he has formulated any opinion as to the cause of the result which he obtained.

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[1] Engineering Dept., The Timken Roller Bearing Co., Canton, Ohio.

[2] R. E. Peterson, *Transactions*, Am. Soc. Mechanical Engrs., APM 55-19, Vol. 1, No. 4, p. 157 (1933).

A. Thum and W. Buchmann, "Dauerfestigkeit und Konstruktion," Vereines deutscher Ingenieure Verlag, Berlin (1932).

[3] Professor of Mechanics, Engineering College, University of Wisconsin, Madison, Wis.

It may be pointed out that of the seven materials reported in the paper, brass was the most sensitive to speed effect, giving an endurance limit at 30,000 r.p.m. which was 35 per cent higher than at 1500 r.p.m.

MR. R. R. MOORE[1] (*by letter*).—By adhering to the fundamental type of test, Mr. Krouse has successfully avoided introducing a number of unknown factors such as often accompany attempts at short-time fatigue tests. Although the conclusions stated at the end of this paper imply that the effect of the speed of the test is not appreciable, a careful study of Table I indicates otherwise.

The tests on brass show that the endurance limit has increased from 20,000 lb. per sq. in. at 1500 r.p.m. to 24,000 lb. per sq. in. at 10,000 r.p.m. which amounts to a 20 per cent increase. The same material shows that at 30,000 r.p.m. the endurance limit has increased 35 per cent. These variations are indeed appreciable. Furthermore the stainless steel tests show an increase of 15 per cent at 30,000 r.p.m. These results are evidence that speeds greater than 10,000 r.p.m. cannot be accepted for endurance tests without further investigation, if such test results are to be used for comparison with results obtained to date. To be specific, it is really important to know whether the endurance limit of this stainless steel is nearer to 60,000 or to 69,000 lb. per sq. in.

A most striking result is brought out in the tests on gray cast iron, which shows a greater endurance limit for the notched specimen than for the polished specimens. In view of the fact that the test diameters of these specimens are very small, it would be valuable to know the size of the cast bars from which they were taken and the location of each type of specimen in the casting. If the casting was of any considerable dimension, there may have been sufficient difference in properties to account for these unusual results.

MR. R. L. TEMPLIN[2] (*presented in written form*).—Probably all investigators of the fatigue properties of metals are continually hoping that sooner or later a testing method will be evolved which involves less time and less expense for determining the endurance limits of such materials. I wish to compliment the author of this paper on the very constructive suggestion he has made in attempting to attain this end.

The data given in Table I, taken as a whole and qualitatively, appear to justify the conclusions he has reached. Quantitatively, however, and with specific reference to that given for duralumin, certain discrepancies may be noted between the values he has given and those obtained in the Aluminum Research Laboratories and presented before this Society last year.[1] On the basis of 50,000,000 cycles at a speed of 3500 r.p.m., we found an endurance limit of about 19,000 lb. per sq. in. as compared to the 15,000 lb. per sq. in. given by the author, for the two speeds of 1500 and 10,000 r.p.m. respectively. In our notch tests, on the basis of 50,000,000 cycles, we find an endurance limit of 9500 lb. per sq. in. representing a decrease in fatigue limit of about 50 per cent, whereas the author found a decrease of only 20 per cent. On the basis of 500,000,000 cycles, we find a reduction of about 40 per cent. The notch which we use is 0.075 in. deep and has a somewhat sharper radius. These facts would appear to indicate that the size and radius of notch is perhaps somewhat more important than he has indicated. The results we have obtained would also put duralumin more nearly in agreement with the other wrought metals which were tested by the author with a possible exception of the S.A.E. No. 1020 steel.

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[1] Consulting Metallurgist, Grantwood, N. J.

[2] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

MR. H. F. MOORE.[2]—I should like to call attention to another high-speed machine which Mr. Krouse has designed and built, which uses an induction motor and a pair of multiplying friction cones to get speeds of 12,000 r.p.m. These friction cones run immersed in light oil.

I should like to call attention also to yet another high-speed machine which uses a series wound commutator motor as direct drive for the specimen. A vacuum cleaner motor has been used in each of two machines now running at the University of Illinois Materials Testing Laboratory. These two machines are giving good service, but the question of how often the brushes will have to be renewed and how often the commutator will have to be turned down is not yet settled.

In all these high-speed machines I would call attention to the relative inexpensiveness of the specimen used.

MR. G. N. KROUSE.[3]—We have taken pains to compute the section modulus on the basis of the diameter at the root of the notch, and we have used that technique on all of these tests, and we believe that this increased endurance limit for cast iron is a property of the material.

J. B. Kommers has found, for a square notched specimen, that the endurance limit was slightly higher. We note this with a good deal of satisfaction. It may be a property of the metal rather than a fault of the test.

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[1] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 364 (1933).

[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[3] Special Research Assistant, Materials Testing Laboratory, University of Illinois, Urbana, Ill.

Mr. Templin's statement that the endurance limit of the metal as he has determined it at 50,000,000 cycles indicated the endurance limit to be 19,000 lb. per sq. in. against the 15,000 lb. per sq. in. reported in the paper may be explained as the rate of failure; that is, for the small specimen, the rate of failure may be higher. The depth of notch which he has used was 0.075 in., and may indicate an increased stress concentration factor reducing the endurance limit to 9500 lb. per sq. in. We should like to make comparative tests on the same bar of material to insure that the material has been aged in the same way.

(*Author's closure by letter*).—The function of this paper is not to foster a particular machine or to state specifically the endurance limit and other properties of any particular metal, but to show in a general way, that the endurance limit of a metal may be determined with less expense and time by using higher testing speeds and simpler test specimens. The fact that two laboratories, testing under similar conditions, do not check within 25 per cent seems to indicate a difference in other properties of the metal, which a later investigation by the author substantiates.

The author does not advocate the use of the maximum speed of 30,000 r.p.m., but does advocate an increase of testing speeds for rotating-beam machines to around 10,000 r.p.m. and a simplification of the test specimen. The use of the V-notch specimen shows that more important practical factors enter into the selection of a design stress where repeated stress governs. This particular type of notch may not cause the worst stress concentration that could exist in some particular structure, but does show that the susceptibility of metals to stress concentrations varies over a wide range.

The economy of higher speed repeated stress testing using simpler test specimens warrants thoughtful consideration.

NOTE ON FRICTIONAL RESISTANCE OF STEEL AND BRASS IN SHRINK FITS[1]

By W. H. Swanger[2]

Synopsis

The making of shrink fits by refrigerating the inner member offers an easily accomplished alternative when the converse method of expanding the outer member by heat is not practicable or permissible. This paper presents data on the resistance to axial slip developed between cylindrical rings 1 in. long and 1 in. in internal diameter assembled on pins which at room temperature were about 0.0015 in. larger in diameter. Prior to assembly the pins were contracted by cooling to −80 C. (−112 F.) in a bath of acetone containing dry ice. Three combinations of material were used: namely, a brass ring on a brass pin; a steel ring on a brass pin; and a steel ring on a steel pin. The resistance to slip was in part dependent upon the amount of "oversize" of the pin but was considerably increased when seizing occurred between the contacting surfaces.

Introduction

It has long been known that considerable holding power is developed by a shrink fit in which a collar or ring is expanded by heat sufficiently to slip over a shaft or spindle which is from 0.001 to 0.002 in. per inch of diameter larger than the inside diameter of the ring at room temperature. When it is either not practicable or permissible to heat the ring, satisfactory shrink fits can be made by the converse method of cooling the pin or shaft. The use of liquid air as the cooling medium, described by Whittemore in 1922,[3] is limited in commercial application by the difficulties in obtaining and handling large quantities of liquid air. The use of solid carbon dioxide as a conveniently transported refrigerant has become common within recent years. By adding "dry ice" to a bath of acetone or other suitable liquid, temperatures of −70 to −80 C. (−94 to −112 F.) can readily be attained. Cooling a shaft of brass from 20 C. (68 F.) to this temperature will decrease its diameter approximately 0.0018 in. per inch of diameter. This is enough to permit the assembly of a cooled shaft into a ring which is sufficiently smaller than the shaft

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Metallurgist, National Bureau of Standards, Washington, D. C.

[3] H. L. Whittemore, "Experimental Use of Liquid Air and Explosives for Tightening Body-Bound Bolts," *American Machinist*, Vol. 56, p. 524 (1922).

when both are at room temperature so that holding power adequate for many purposes is obtained after the shaft has warmed up.

A material having a much smaller coefficient of expansion than brass, steel for example, may not shrink enough in dimensions when cooled in a bath of "dry ice" to make a tight fit in this manner. The difficulty frequently can be overcome by warming the outer member in hot water before attempting to slip it over the cooled shaft.

The resistance to slip between a shaft and encircling ring, assembled by a shrink fit, is dependent only partly upon the amount by which the shaft diameter at room temperature exceeds the diameter of the hole; it may also be influenced decidedly by the extent to which seizing takes place on the two metal surfaces in close contact. Seizing is more likely to occur in a shrink fit made by refrigerating the inner member than in one made by expanding the outer member by heat, if, in the latter case an oxidized surface tending to inhibit seizing is formed on the heated member. It is well known, however, that materials differ decidedly in their tendency to seize. This is clearly shown by the experiments described below in which the resistance to slip under tension was measured on shrink fits between brass on brass, brass on steel, and steel on steel.

TABLE I.—DIMENSIONS[a] OF PINS AND RINGS AND FORCE NECESSARY TO SEPARATE THEM.

	Diameter at 20 C. (68 F.), in.		Oversize of Pin at 20 C. (68 F.), in.	Diameter of Pin at −80 C. (−112 F.), Calculated, in.	Diameter of Ring at 100 C. (212 F.), Calculated, in.	Diametral Clearance When Assembled, Calculated, in.	Resistance to Initial Slip, lb. per sq. in. of contact area	Maximum Resistance Developed, lb. per sq. in. of contact area
	Pin	Ring						
Brass ring on brass pin.	1.0009	0.99964	0.00126	0.9991	....	0.00054	722	1430
Steel ring on brass pin.	1.00211	1.00075	0.00136	1.00031	1.00163	0.00132	1700	2100
Steel ring on steel pin..	1.00225	1.00081	0.00144	1.00115	1.00169	0.00054	1820	6050

[a] The measurements were made by the Gage Standardization Section of the Bureau of Standards. The diameters of the pins were measured with a Pratt and Whitney measuring machine. The internal diameters of the rings were measured with a John Bath internal micrometer.

PREPARATION OF SPECIMENS AND TESTS

Cylindrical pins approximately 1 in. in diameter were machined from 2-in. bars of yellow brass and machinery steel. The composition of the brass was: 61.3 per cent copper, 2.9 per cent lead, less than 0.1 per cent iron, and, by difference, 35.7 per cent zinc. The brass had a Brinell hardness number, 10-mm. ball, 500-kg. load, of 90; and a Rockwell hardness number, B scale, $\frac{1}{16}$-in. ball, 100-kg. load, of 54. The brass had a duplex or α-β structure with lead disseminated in fine particles throughout (Fig. 6). The steel contained 0.12 per cent carbon and 0.14 per cent sulfur. The steel had a Brinell hardness number, 10-mm. ball, 3000-kg. load, of 187; and a Rockwell hardness number, B scale, $\frac{1}{16}$-in. ball, 100-kg. load, of 90.

One end of each pin was left at the original diameter of the bar so as to form a head. The other end was carefully ground so that it was truly cylindrical to within less than 0.0001 in. over a length of about 2 in. from the end. Cylindrical rings slightly longer than 1 in. were prepared from the same materials with holes approximately 1 in. in diameter. They were slightly bell mouthed at each end and were carefully reamed to be truly cylindrical to within less than 0.0001 in. over a length of 1 in. The diameter of each pin at room temperature was about 0.0015 in. larger than the inside diameter of the corresponding ring which was to be slipped over the pin. The diameters measured at 20 C. (68 F.) with an accuracy of ±0.00003 in. for the pins and ±0.00005 in. for the holes are given in Table I.

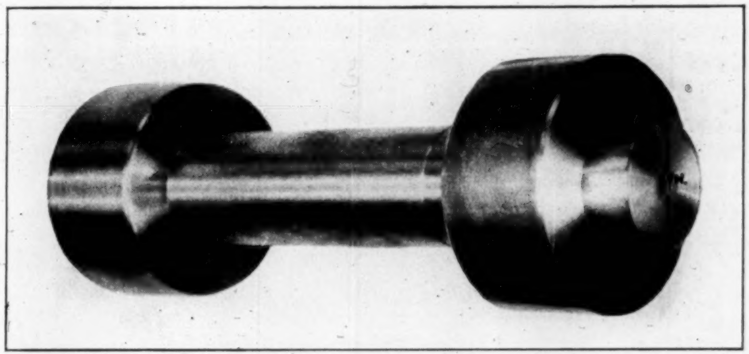


FIG. 1.—Assembled Pin and Ring, Brass.

Each pin was immersed in a bath of acetone containing solid carbon dioxide until it attained temperature equilibrium with the bath, at −80 C. (−112 F.). The reduction in diameter of the pins was calculated for a decrease in temperature of 100 C. (180 F.) on the basis of temperature coefficients of expansion of 0.000018 in. per in. per deg. Cent. (0.000010 in. per in. per deg. Fahr.) for brass, and 0.000011 in. per in. per deg. Cent. (0.000006 in. per in. per deg. Fahr.) for steel. These are average values at room temperature, considered to be sufficiently accurate for the purpose, and the very slight decrease for lower temperatures was not taken into account.

The brass ring was slipped on the cooled brass pin without difficulty. A film of acetone probably adhered to the pin and may have acted as a lubricant and aided quick assembly. The contraction

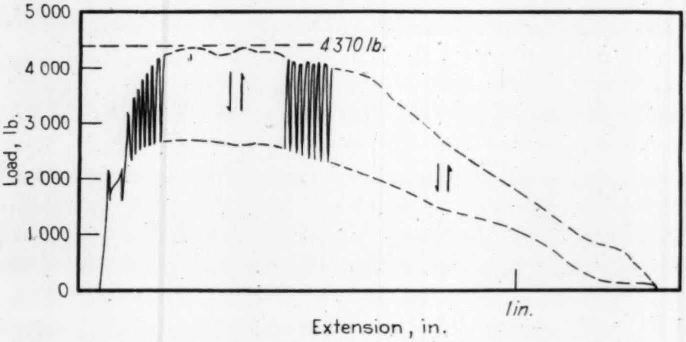


FIG. 2.—Load-Slip Diagram, Brass Ring Pulled from Brass Pin.

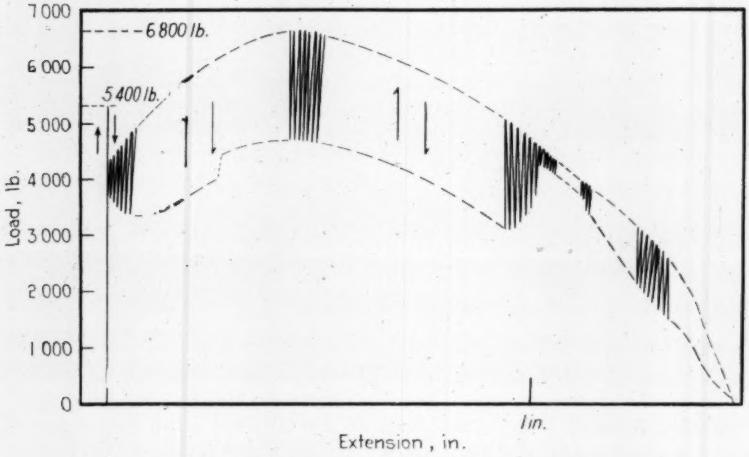


FIG. 3.—Load-Slip Diagram, Steel Ring Pulled from Brass Pin.

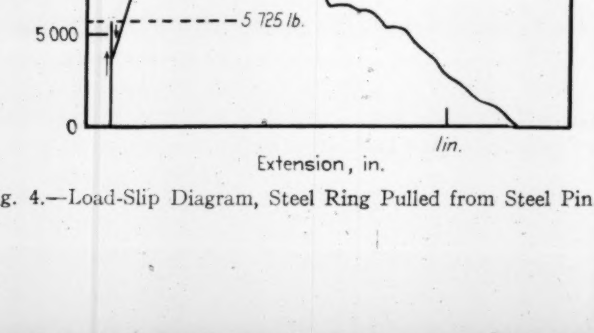
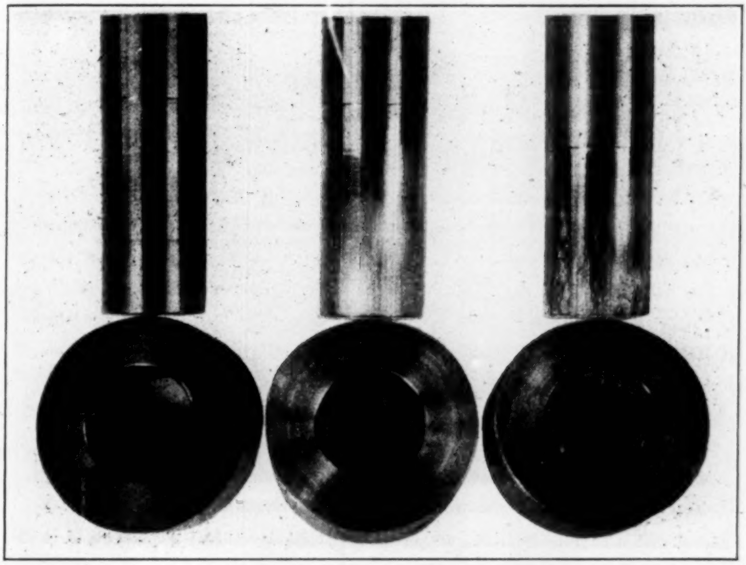


Fig. 4.—Load-Slip Diagram, Steel Ring Pulled from Steel Pin.

in the steel pin was not sufficient to permit its insertion into the steel ring, and the latter was heated in boiling water to obtain sufficient clearance for easy assembly. This was also found necessary in order to slip a steel ring on the cooled brass pin. An assembled pin and ring are shown in Fig. 1.

After the assembled unit had come to room temperature, the ring was pulled from the pin in a hydraulic type of tension testing machine. Automatically recorded load-slip diagrams are reproduced in Figs. 2, 3, and 4. The appearance of the units after the rings were separated from the pins is shown in Fig. 5.



(a) Brass ring on brass pin. (b) Steel ring on brass pin. (c) Steel ring on steel pin.

FIG. 5.—Disassembled Pins and Rings.

Brass Ring on Brass Pin:

The brass ring on the brass pin supported a load of 2200 lb. (722 lb. per sq. in. of contact area) before initial slip occurred. The load necessary to continue slippage then increased rather rapidly with slip taking place in small increments until a maximum of 4370 lb. was reached (1430 lb. per sq. in. of contact area). Continued slip in small increments then proceeded at decreasing loads until the ring cleared the end of the pin. The appearance of the pin after removal of the ring (Fig. 5) indicated that no appreciable amount of visible seizing had taken place. Most of the increase in load after initial slip was probably absorbed in causing elastic deformation in pin and ring as the ring was forced over the protruding end of the pin. No relative change in dimensions was found after the two had been separated. The loading mechanism of the testing machine was such that when a small amount of slip occurred the load immediately decreased until slip halted, which was approximately at the load at which initial slip took place. This is believed to account for the broad band of load fluctuation shown in Fig. 2.

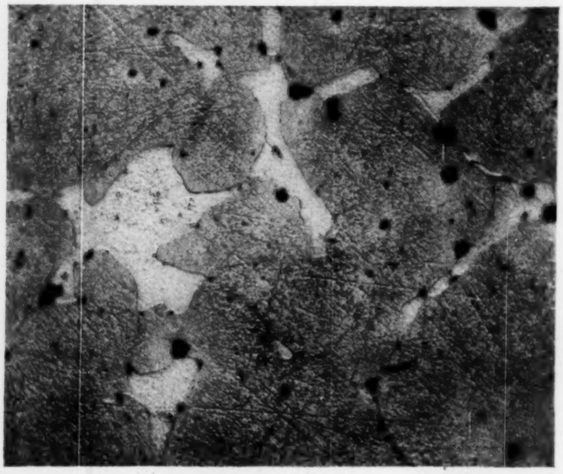


FIG. 6.—Microstructure of the Brass (× 500).

Etched with a mixture containing 86 per cent of strong ammonium hydroxide and 14 per cent hydrogen peroxide.

Steel Ring on Brass Pin:

The steel ring on the brass pin supported a load of 5400 lb. (1700 lb. per sq. in. of contact area) when initial slip took place. The load immediately decreased. Slip then continued in increments somewhat smaller than in the all-brass assembly and at increasing loads until a maximum of 6800 lb. was reached (2100 lb. per sq. in. of contact area). The band of load fluctuation on the load-slip diagram, Fig. 3, was somewhat similar to that obtained with the all-brass unit. The diagrams, as reproduced in Figs. 2 and 3, do not show all of the lines drawn by the automatic load recording device but show the maxima and minima of the load fluctuations.

The brass pin was distinctly scored by the steel ring as is shown by the photograph, Fig. 5. Evidently seizing had taken place to a greater extent between the steel and brass than between the brass ring and pin. This seizing was probably responsible for the greater holding power of the steel ring on the brass pin, although the slightly larger amount of over-size of the pin with respect to the ring may also have contributed somewhat.

Steel Ring on Steel Pin:

The steel ring on the steel pin did not slip initially until a load of 5725 lb. was applied (1820 lb. per sq. in. of contact area). As in the two previous cases, the load immediately decreased but then increased rapidly, without any slip by increments and resultant intermediate decreases in load, until a maximum of 19,000 lb. was reached (6050 lb. per sq. in. of contact area).

Severe seizing had taken place between the ring and pin as is shown in Fig. 5. Portions of metal had been torn bodily from the pin where the adhesive force between the contact surfaces of pin and ring exceeded the cohesive strength of the steel. The forces necessary to rupture the metal were evidently so far in excess of those required to cause slip on the contact area where seizing had not occurred, that the load-slip diagram obtained, Fig. 4, was essentially similar in form to a load-extension diagram obtained normally in rupturing a tension test specimen of a ductile metal.

SUMMARY

The experiments described indicate that in shrink fits made by refrigerating the inner member, seizing does not occur until relative motion has taken place between the two contacting surfaces. The force necessary to cause initial slip is probably largely dependent upon the amount of "over-size" of the inner member. After seizing has occurred, the force necessary to continue the separation of the fitted parts is very much increased.

It is believed that the difference in behavior of the brass and the steel assemblies is characteristic of these two classes of materials in general. The failure of the brass to seize is undoubtedly to be associated with the particles of metallic lead disseminated throughout the alloy whereas the presence of the brittle sulfide particles in the steel is responsible, at least in part, for the tendency of the steel to score the brass.

DISCUSSION

Mr. F. V. Hartman[1] (*presented in written form*).—Mr. Swanger's results are very interesting. The method he uses to make shrink fits by cooling the inner member to shrink it so that it may then be expanded into the outer member has been used successfully in a number of instances. Reference is made to the fitting of aluminum parts where heating may sometimes be undesirable.

According to Table I, the brass pin which was slipped into the brass ring was 0.00126 in. oversize at 20 C., and the brass pin that was slipped into the steel ring was 0.00136 in. oversize. We are wondering whether this difference in oversize is sufficient to explain the fact that the first brass pin could be slipped into the brass ring satisfactorily, whereas the second brass pin could not be contracted sufficiently to permit its insertion into the steel ring, necessitating expansion of the steel ring by heating it in boiling water. Although the difference is small, this may be the explanation, because if the dimensions of the two parts in each of these two assemblies were identical there seems no reason why the contracted brass ring should not slip into a steel ring just as easily as it would into a brass ring, the rings being both held at 20 C.

In fitting aluminum parts together, experience indicates that a greater diametral clearance between the parts during assembly is desirable, and in fact practically necessary, in order to get the parts together satisfactorily. It is also important to make provision for slipping the ring over the pin very rapidly. If accurate alignment of the two parts is not provided for, and if the parts are not slipped together rapidly, sufficient contact between the two parts may be made to cause them to "freeze" together before the parts are completely assembled. This is explained by the high heat conductivity of aluminum which causes a rapid heat transfer between the two parts when they touch and may cause a sufficient loss of the diametral clearance to prevent fitting the ring completely over the pin.

It seems that Mr. Swanger might have explained some of the results more satisfactorily. No doubt, as he has stated, the difference in behavior of the brass and steel assemblies is characteristic of these two classes of materials. However, in making this statement, it seems that more consideration should be given to the probable surface pressures between the pin and the encircling ring. There is no question but what seizing is largely a characteristic of the material, but, on the other hand, seizing also depends upon the pressure between the surfaces that are rubbing together.

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[1] Research Mechanical Engineer, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

He also states that the force necessary to cause initial slip is probably largely dependent upon the amount of "oversize" of the inner member. In connection with this statement, it seems that consideration of the probable surface pressures should again be taken into account. The probable surface pressures between the different assemblies tested depend on the moduli of elasticity of the materials as well as on the amount of oversize of the inner member.

Taking 29,000,000 lb. per sq. in. for the modulus of elasticity of the steel and 15,000,000 lb. per sq. in. for brass, it follows that the pressure between the surfaces will be about twice as much for a steel ring on a steel pin as for a brass ring on a brass pin, assuming that the oversize is the same in both cases. It also follows that the pressure between the surfaces for a steel ring on a brass pin would probably be somewhere in the neighborhood of 50 per cent more than for a brass ring on a brass pin. From these two statements and in view of the amount of oversize of the pin at 20 C., it is quite evident that the surface pressure was considerably higher in the case of the steel on steel assembly than either of the other two assemblies, while the surface pressure of the brass on brass assembly is by far the lowest of the three.

The removal of the ring from the pin is mostly a coefficient of friction problem wherein the initial load required to start movement is dependent upon the pressure between the surfaces and the static coefficient of friction. This seems to help considerably in the explanation of the loads required to cause initial slip, since the discussion in the above paragraph at least gives an idea of the probable order of surface pressure that existed, even though we do not know the static coefficient of friction values. After movement once has started, seizing may or may not occur, depending upon the characteristics of the materials and also upon the surface pressures.

It is, therefore, possible that seizing would also have occurred in the first assembly with the brass ring on the brass pin had the pin been sufficiently oversize to cause higher surface pressures. Conversely, seizing might not have occurred on the second and third assemblies had the surface pressures in these been as low as in the first. It is, therefore, suggested that if the tests were repeated quite different results would be observed if the amount of oversize of the pin is made such as to give more nearly equal surface pressures. It would also seem desirable in repeating this work to test a number of specimens of each different kind to see how they check each other.

It seems reasonable that the amount of oversize to allow for the inner member of shrink fit assemblies should be determined largely by the modulus of elasticity of the materials. For instance, if an allowance of 0.001 in. per in. of diameter is considered satisfactory for a steel pin in a steel ring, the allowance should be about double this amount for a brass pin in a brass ring and three times this amount for an aluminum pin in an aluminum ring. The results of Mr. Swanger's tests seem to indicate this and our experiences in fitting aluminum parts together also agree with this assumption.

MESSRS. O. J. HORGER[1] AND J. L. MAULBETSCH[1] (*presented in written form*).—Since it is more general to assemble tight-fitted parts by the application of heat or use of pressure than through the use of coolants, it would appear worth while to have comparative data like the author's Figs. 2, 3, 4 and 5 for the more usual methods of assembly mentioned above. This comparison may indicate some advantages or otherwise of one method of assembly over another.

Is the author in a position to state whether, at equal press fit pressure, brass will not seize and steel will because of the difference in their characteristics as explained in the summary of the author? In the three cases studied by the author the dimensions of the press fit were approximately the same, but due to the difference in the modulus of elasticity for steel and brass, the press fit pressure in the first test will be much less than the pressure in the third test, the second will be intermediate. The writers would like to know what effect the absolute value of the press fit pressure would have on the seizing effect.

Furthermore, with the conditions of assembly outlined, the shrink fit pressure at the end of the sleeve is considerably higher than between the ends. This "end effect" may account to some extent for the seizing observed. A relieved end construction on the sleeve may reduce this seizing and we should like to ask if the author has given any consideration to this aspect.

MR. W. H. SWANGER[2] (*author's closure*).—Mr. Hartman postuates that if the oversize of the brass pin assembled into the steel ring had been the same as in the brass pin—brass ring assembly, it would have been possible to insert the cooled brass pin into the steel ring when the latter was at room temperature. I am certain this would have been the case. However, after one failure to make the assembly with the slightly smaller clearance under these conditions, possibly because of "fumbling" the operation, the steel ring was warmed as stated, so that the assembly was easier to make. The conditions after assembly would have been the same in either case. I also agree with Mr. Hartman that the tendency for one metal to seize upon another is largely an inherent characteristic of the metals concerned, and that the occurrence of seizing depends upon the pressure at the contacting surfaces. For a given oversize of pin, with respect to the inner diameter of an encircling cylinder, the pressure at the contacting surfaces is dependent upon Young's modulus of elasticity of the material, as was stated by Mr. Hartman, but, further, is also influenced by (*a*) the cubical modulus of compressibility in which Poisson's ratio is a factor, (*b*) the ratio of length to diameter in the contacting parts, and (*c*) the strength of the material. This last factor means that pressure at the contacting surfaces increases with increase in oversize or increase in Young's modulus only within the range where negligible permanent set is produced. The "end effect" mentioned by Messrs. Horger and Maulbetsch is involved in factor (*b*) just mentioned.

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[1] Engineering Dept., The Timken Roller Bearing Co., Canton, Ohio.

[2] Metallurgist, National Bureau of Standards, Washington, D. C.

An analysis of each of these factors and experimental data to illustrate them are beyond the scope of this paper which was presented to show that a practical application can be made of "dry ice" refrigerant to assemble parts by shrink fits.

It is regretted that there was no opportunity to make comparisons between shrink fits produced as described in the paper and press fits or shrink fits produced by expanding the outer member by heating it. An excellent discussion on this subject is given in a very recent paper entitled "Factors Affecting the Grip in Force, Shrink, and Expansion Fits" by Robert Russell.[1]

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[1] *Proceedings*, Inst. Mechanical Engrs., Vol 125, p. 493 (1934).

THE IMPACT STRENGTH OF COMMERCIAL ZINC ALLOY DIE CASTINGS

BY E. A. ANDERSON[1] AND G. L. WERLEY[1]

SYNOPSIS

Charpy impact test specimens, 525 in number, were cut from 77 commercial zinc-alloy die castings and subjected to impact tests in comparison with test bars die cast to the same size. All of the test specimens were so cut as to leave one unmachined die-cast face. In conducting the Charpy impact test, the specimens were placed in the machine in such a manner as to place this die-cast face under tension. The results obtained indicate a close agreement between the impact strength of the machined specimens and of specimens die cast to size. The authors draw no conclusion relating to the correlation of these values to the over-all strength of the original die casting.

The Society's Tentative Specifications for Zinc-Base Alloy Die Castings (B 86 – 33 T)[2] describe the physical properties to be expected of zinc-alloy die castings of two specific compositions on the basis of tests on specimens cast to size. The question has been raised on many occasions as to whether the properties of commercial die castings approximate at all closely those of test specimens cast to size. The authors felt that a study of this point was highly desirable.

Charpy impact tests were carried out on some 520 test specimens machined from 77 commercial die castings. Since most of the castings obtained for this work were less than ¼ in. in thickness, it was necessary to establish new specimen sizes for the thinner sections. Data on these new standards have been established using specimens cast under the same controlled conditions used in the preparation of the conventional ¼-in. square test specimens.

All of the specimens tested were cut in such a manner as to include one unmachined die-cast face and the impact tests were so conducted as to place this face in tension in every instance.

AVAILABLE CHARPY IMPACT STRENGTH DATA FOR TEST BARS CAST TO SIZE

The Tentative Specifications B 86 – 33 T state the following minimum initial physical properties for test bars die cast to size:

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[1] Chief Investigator, and Investigator, respectively, Metal Section, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 644 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 257.

	AVERAGE OF FIVE SPECIMENS		INDIVIDUAL SPECIMENS	
	ALLOY NO. XXI[c]	ALLOY NO. XXIII[c]	ALLOY NO. XXI	ALLOY NO. XXIII
Tensile strength, lb. per sq. in.[a]	44 000	35 000	35 000	30 000
Elongation in 2 in., per cent[b]	2.0	3.0	0.5	2.0
Charpy impact, ft-lb.[b]	6.0	12.0	4.0	8.0

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[a] ¼-in. round section.

[b] ¼ by ¼-in. section.

[c] The alloys shall conform to the following compositions:

	ALLOY NO. XXI	ALLOY NO. XXIII
Aluminum, per cent	3.5 to 4.5	3.5 to 4.5
Copper, per cent	2.5 to 3.5	0.1 max.
Magnesium, per cent	0.02 to 0.10	0.03 to 0.08
Lead, max., per cent	0.007	0.007
Cadmium, max., per cent	0.005	0.005
Tin, max., per cent	0.005	0.005
Iron, max., per cent	0.100	0.100
Zinc, per cent	remainder	remainder

The authors have published, at various times, data on these same alloys as follows:

	ALLOY NO. XXI			ALLOY NO. XXIII		
	MAXIMUM	MINIMUM	AVERAGE	MAXIMUM	MINIMUM	AVERAGE
Tensile strength, lb. per sq. in.	52 800	45 700	47 900	44 600	36 600	40 300
Elongation in 2 in., per cent	7.5	3.1	5.1	7.3	2.0[a]	4.7
Charpy impact, ft-lb.[b]	23.25	16.50	19.00	34.75	14.00	20.00

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[a] Lower than A.S.T.M. minimum but exceeded A.S.T.M. minimum after short aging period.

[b] The initial impact strength of these alloys varies with the magnesium content, being highest when the magnesium is at the minimum value. The authors' data were obtained from alloys containing about 0.04 per cent of magnesium whereas the A.S.T.M. specification minima are based on the possibility of having the maximum magnesium content present. It may be pointed out that commercial die castings are usually made with magnesium contents near the minima permitted by the specifications and hence should tend to approximate the authors' impact strength data more closely than the specification requirements.

The maximum and minimum values given above are averages of results obtained on ten specimens each. The average data represent the average of results from ten specimens each of seven separate casts in the case of alloy No. XXI and of thirteen separate casts in the case of alloy No. XXIII. The specimens used were die cast by the authors in a commercial die-casting machine and fulfilled the test bar specification requirements as to dimensions.

Die Castings Used:

A total of 77 commercial die castings ranging in weight from 3 oz. to 13½ lb. and in length from 3 in. to 49 in. were obtained from various commercial die casters. The castings were selected at random from regular production and in no case represented special casting conditions.

From the castings, 520 impact test specimens were cut and tested. Since the castings represented a wide variety of shapes and sections it is felt that the data obtained furnish a reasonably accurate representation of the impact properties of commercial die castings.

All of the castings obtained were made either of alloy No. XXI or of No. XXIII. Since the difference in impact strength between these alloys is not significant when first cast, no attempt has been made to distinguish between them in the present discussion. Had the tests been made on castings a year or two old the characteristic loss in impact strength of alloy No. XXI would have required consideration.

Preparation of Specimens:

The most desirable specimen would be one 3 in. long with a $\frac{1}{4}$-in. square section. This would correspond to the standard A.S.T.M. impact test specimen. While a number of castings were obtained from which such specimens could be and were machined, it was realized that the majority of die castings today are less than $\frac{1}{4}$ in. in thickness and could not be investigated by means of the standard specimen.

In order to cover the widest possible range of commercial castings in this survey, the following specimen sizes were selected:

LENGTH, IN.	WIDTH, IN.	THICKNESS, IN.
3	$\frac{1}{4}$	$\frac{1}{4}$
3	$\frac{1}{2}$	$\frac{1}{8}$
3	$\frac{1}{2}$	0.080
3	$\frac{1}{8}$	$\frac{1}{8}$

In the selection of test areas from a given casting every effort was made to obtain the largest possible of the above sizes. Thus the use of a specimen $\frac{1}{2}$ by 0.080 in. implies that the actual section thickness of the casting was less than $\frac{1}{8}$ in. or else the $\frac{1}{2}$ by $\frac{1}{8}$-in. specimen would have been prepared.

In the machining of the test specimens, the practice was established of leaving one face with the original die-cast surface undisturbed. In the impact test this face was always turned away from the hammer in the position to take the maximum tensile deformation.

In the case of the $\frac{1}{2}$ by $\frac{1}{8}$-in. and the $\frac{1}{2}$ by 0.080-in. specimens, the face left unmachined was always that having the dimensions $\frac{1}{2}$ by 3 in.

Test Bar Specimens:

Data were already available on $\frac{1}{4}$-in. square cast test bars, as given above, for comparison with the machined bars. In order to obtain some idea of the agreement to be expected between cast bars and machined specimens, some of the other shapes were supplied in both forms. Data on these specimens will be found in Table I.

Method of Testing:

The machine used was of the Charpy type having a potential breaking force of 36.75 ft-lb. The specimens were placed on a 40-mm. span in such a position as to place the die-cast surface away from the striking tup. It is estimated that the values obtained are accurate to the nearest 0.25 ft-lb.

Results Obtained:

The data obtained in these tests are presented in the form of frequency graphs. Figure 1 shows such a graph for the $\frac{1}{4}$ by $\frac{1}{4}$-in. specimens. Only those test specimens cut from commercial castings are included in the charts.

TABLE I.—IMPACT STRENGTH OF TEST BARS, CAST TO SIZE OR MACHINED TO SIZE, AS INDICATED.

Size of Specimen, in.	Type of Specimen	Number of Bars of Each Alloy	Impact Strength, ft-lb.	
			Alloy No. XXI	Alloy No. XXIII
$\frac{1}{8}$ by $\frac{1}{8}$	Cast to size	65	3.50	4.00[a]
$\frac{1}{8}$ by $\frac{1}{8}$	Machined from $\frac{1}{4}$ by $\frac{1}{4}$-in. cast bars	26	3.00	4.00[a]
$\frac{1}{8}$ by $\frac{1}{8}$	Machined from $\frac{1}{2}$ by $\frac{1}{2}$-in. cast bars	16	3.50	3.50
$\frac{1}{2}$ by $\frac{1}{8}$	Cast to size	58	16.50	19.75[a]
$\frac{1}{2}$ by $\frac{1}{8}$	Machined from $\frac{1}{2}$ by $\frac{1}{2}$-in. cast bars	26	16.50	15.00
$\frac{1}{2}$ by 0.080	Machined from $\frac{1}{2}$ by $\frac{1}{8}$-in. cast bars	35	9.50[a]	7.75[a]
$\frac{1}{2}$ by 0.080	Machined from $\frac{1}{2}$ by $\frac{1}{2}$-in. cast bars	15	9.00[a]	7.50[a]

[a] Some of these specimens did not break under the impact load but bent through the 40-mm. opening of the machine.

The peak of the curve falls between 10 and 20 ft-lb. The average, 18.75 ft-lb., agrees well with the authors' average test bar data of 19.00 ft-lb. for alloy No. XXI and 20.00 ft-lb. for alloy No. XXIII.

Attention is called to the relatively large number of specimens which exceeded 35 ft-lb. in strength. These were all cut from a group of automobile windshield stanchions of heavy section weighing about 10 lb. each. It would seem that for a depth of $\frac{1}{4}$ in., at least, large-section castings can be at least as strong as test bars.

The few specimens showing low strength were cut from specific areas of castings showing excellent strength in other areas. The values obtained were low because of porosity of a type which can readily be overcome by comparatively slight changes in the venting of the die.

The data from the $\frac{1}{8}$ by $\frac{1}{8}$-in. bars are plotted in Fig. 2. It is seen at once that the great majority of the test specimens fell in the region

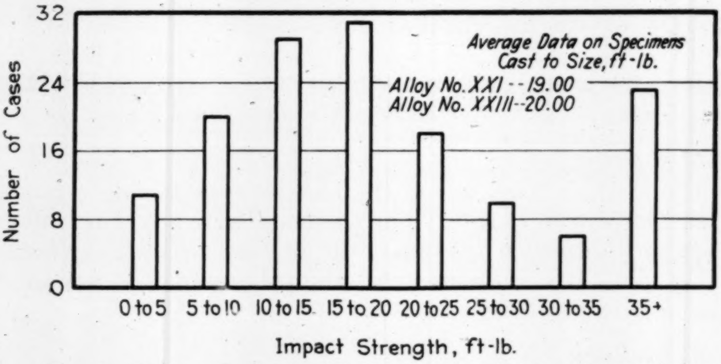

FIG. 1.—Impact Frequency Graph, ¼ by ¼-in. Impact Bars Cut from Commercial Castings.

Average Impact Strength of 148 Bars—18.75 ft-lb.

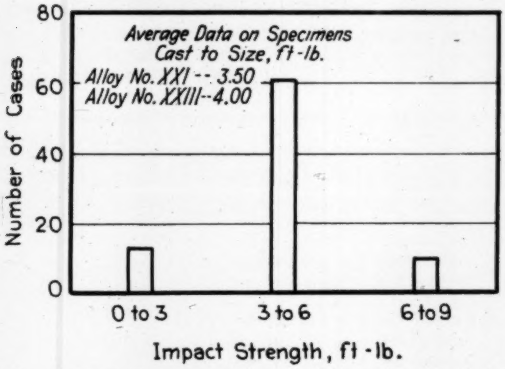

FIG. 2.—Impact Frequency Graph, ⅛ by ⅛-in. Impact Bars Cut from Commercial Castings.

Average Impact Strength of 84 Bars—4.50 ft-lb.

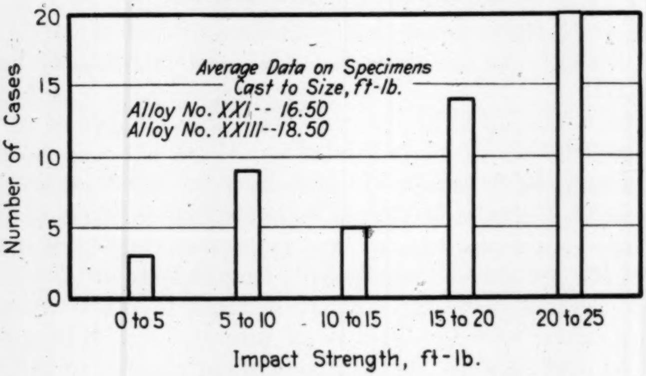

FIG. 3.—Impact Frequency Graph, ½ by ⅛-in. Impact Bars Cut from Commercial Castings.

Average Impact Strength of 51 Bars—16.25 ft-lb.

3 to 6 ft-lb. The average strength of 4.50 ft-lb. is somewhat higher than the values 3.50 and 4.00 ft-lb. reported on test bars of alloys Nos. XXI and XXIII.

The tests on the ½ by ⅛-in. bars furnished the data plotted in Fig. 3. The average impact strength of 16.25 ft-lb. is in close agreement with the values obtained on test bars.

The thinner ½ by 0.080-in. test specimens yielded the data given in Fig. 4. Of the 177 bars tested, 113, or 64 per cent, fell in the region 9 to 12 ft-lb. The average value 8.50 ft-lb. lies between the figure 9.00 for alloy No. XXI and that of 7.75 for alloy No. XXIII.

Discussion of Results:

It is apparent from the data that so far as impact strength is concerned a close agreement exists between the properties of die-cast test bars and of sections cut from the surface of commercial die castings.

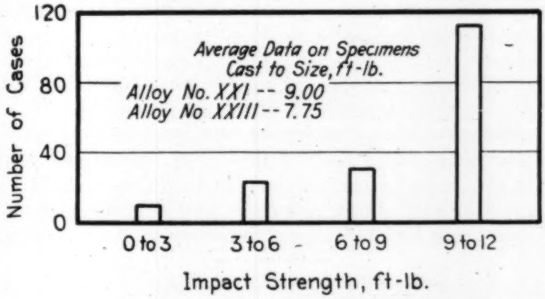

FIG. 4.—Impact Frequency Graph, ½ by 0.80-in. Impact Bars Cut from Commercial Castings.
Average Impact Strength of 177 Bars—8.50 ft-lb.

The authors are not prepared, at the present time, to draw any further conclusions from the data but would point out that with the exception of three large windshield parts the specimens tested represented at least one-half of the total thickness of the castings investigated. No attempt will be made at this point to correlate the results obtained on machined specimens with the practical impact strength of the commercial castings as a whole.

Acknowledgment:

The authors are indebted to Mr. John Craig of this laboratory for his assistance in carrying out the impact tests. Credit is also due the die casters who cooperated fully in the selection of suitable castings for the tests.

SOME FACTORS AFFECTING STRAIN MEASUREMENTS IN TESTS OF METALS

BY R. L. TEMPLIN[1]

SYNOPSIS

This paper is based on results of a large number of tests carried out for the purpose of affording a comparison of deformations on various elements of both tension and compression specimens of various forms in both the elastic and plastic ranges.

The testing procedure has been laid out for the purpose of determining the effects of eccentric and oblique loadings on the specimens; the effects of different types of testing machine jaws on the deformations obtained and the effects of different types of plugs in the case of the hollow round tension specimens.

The results obtained indicate definitely the necessity for uniform and axial loading of specimens, especially when determining the elastic modulus. Under such conditions of testing, it appears relatively unimportant whether strains are measured on one or more elements of the specimen.

Under conditions of non-uniform or non-axial loading, it would appear necessary to determine strains simultaneously on opposite elements of a specimen in order to obtain satisfactory values for modulus of elasticity.

The test conditions for obtaining satisfactory yield strength values are much less exacting than those for obtaining satisfactory modulus values.

There are many factors which affect strain measurements in the testing of metals for mechanical properties. This fact was well recognized sixty or more years ago by those investigators in Germany who carried out much of the early classical work in the materials-testing field. The strain-measuring apparatus and testing machines which these pioneers had to work with may not have had refinements of design and workmanship found in present-day testing equipment but did apparently have sensitivities and accuracy tolerances on a par with the apparatus we are using today. For example, Bauschinger's mirror extensometer, perfected in 1873, was capable of measuring strains to one part in 100,000. Martens' modification of Bauschinger's device of the same degree of precision followed shortly thereafter. Two paragraphs appearing in Martens' Handbook of Testing Materials (1898)[2] are quite indicative of the conclusions reached by the foremost investigators of that time, concerning factors affecting strain measurements in tests of metals.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] Pages 27 and 61.

In 1914 this Society changed its standard methods regarding attachment of extensometers to tension specimens, from "to two sides of the specimen" to "on the two opposite sides of the specimen in the case of flats, and at three equidistant points in the case of rounds." After much discussion, including dissension, the standards of 1916 were adopted. In these appear the requirements that "the extensometer should be attached to at least two sides of the specimen to compensate for unequal elongation, for improper holding, or for any slight bending that may exist in the specimen." These requirements remained in the standards until ten years ago when the standards were discarded and new tentative methods issued which omitted all requirements involving number of points of attachment for extensometers. The new standards adopted last year likewise contain no such requirements.

While some very definite opinions of testing men have been registered throughout this history, yet there seems to be a conspicuous absence of factual data in evidence. Furthermore, today, in routine inspection tension tests of metals, we are more concerned with determinations of yield points and yield strengths than with modulus of elasticity, elastic limit, and proportional limit values. It follows, therefore, that we must give attention to the factors affecting strain measurements with a view towards obtaining a better quantitative appreciation of their effects on the properties just mentioned.

MATERIAL AND SPECIMENS

In the present instance we shall confine our attention to metals having no yield points but for which yield strengths must be determined. The investigational work that will be described was carried out for the purpose of obtaining a better quantitative understanding of the factors affecting strain measurements on both tension and compression specimens of various forms. Tension tests were made on standard A.S.T.M. sheet specimens[1] with various ratios of width to thickness; on standard 0.5-in. diameter round threaded-end specimens[1] under axial, oblique, and eccentric loads; on standard tubular specimens[1] with different ratios of diameter to wall thickness and tested with two types of V-grips using both poorly and well-fitted plugs. Compression tests were made on tubular, angle, channel, and I-beam specimens of short length to show the effects of form of specimen on strain measurements under optimum practical test conditions.

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[1] See Tentative Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8 - 33), 1933 Book of A.S.T.M. Standards, Part I, p. 949.

The metals used for the tests were high-strength wrought aluminum alloys, but quite similar results would be anticipated with any other metals having similar stress-strain characteristics. Such other metals would be cold-worked copper or steel, nickel, alloys of magnesium, stainless steel, and many others.

TESTING PROCEDURE

The tests were all carried out in multi-range Amsler machines using the lowest capacity possible for a given specimen. The accuracy of each range used was well within ±1 per cent. Spherically seated grips were used wherever possible, and for the sheet specimens grips were used which insured both axial and uniform gripping of the test specimens. For the obliquely and eccentrically loaded specimens, the test conditions were purposely exaggerated considerably beyond those normally encountered in present-day laboratories, in order better to illustrate the effects of these factors.

The strain gages or extensometers used for most of the tests were of the 1-in. gage length, Huggenberger type with a nominal magnification ratio of 1200:1. Estimation of readings to the nearest fifth of a division gave a sensitivity with these instruments, of one part in 120,000. For a metal having a modulus of elasticity of about 10,000,000 lb. per sq. in., this sensitivity corresponds to a stress of about 80 lb. per sq. in. For some of the tests a $\frac{1}{2}$-in. gage length type was used which had a nominal magnification ratio of 2000:1 and a sensitivity of one part in 100,000, corresponding to a stress of about 100 lb. per sq. in. Before use in the tests each instrument was carefully calibrated[1] and its multiplication constant determined. These constants were then used in converting instrument readings into strain measurements.

The set value used for yield strength is the one now in general use in this country for non-ferrous metals, that is, 0.2 per cent, as determined in accordance with the standard A.S.T.M. method.

In carrying out the tests, from two to ten strain gages were used simultaneously on a specimen. For the elastic strain measurements, readings were taken at intervals of stress corresponding to nominal values of 3000, 6000, 9000, 12,000, 15,000 and 18,000 lb. per sq. in. The load was then released to some low value between 1000 and 2000 lb. per sq. in.; each gage shifted to the next gage line; the loads and corresponding strain readings repeated; and so on until a series of readings was obtained with each instrument on each gage line. This procedure gave stress-strain data for each instrument used on every gage line in a given test. From such data, variations resulting from both individual instruments and gage-line location could be determined. Also variations in modulus of elasticity values from the grand average value of the specimen were readily determined. When the specimens were stressed beyond the elastic range, each instrument was retained on a specific gage line until after the yield strength was passed. One of the specimens with strain gages attached is shown in Fig. 1.

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[1] R. L. Templin, "The Calibration of Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 714 (1928).

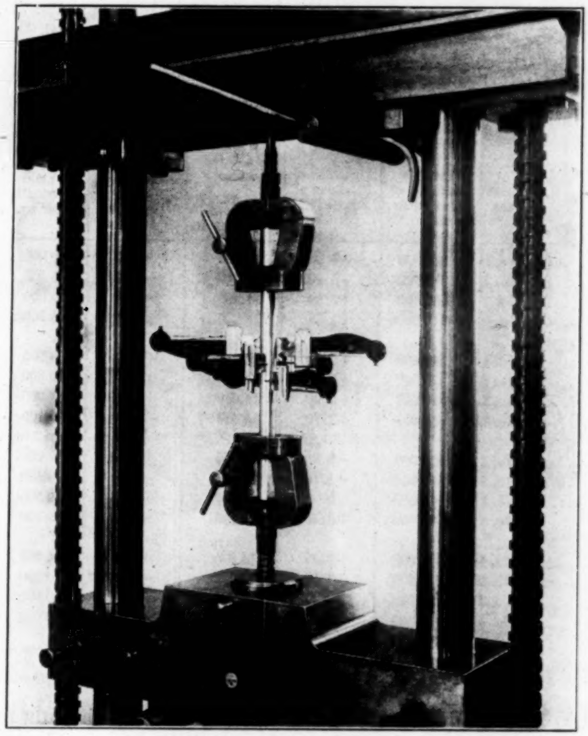

FIG. 1.—Tubular Tension Specimen Ready for Test.

Without exception all of the stress-strain data when plotted gave straight-line relationships, within the sensitivity and observational error limitations of the strain gages, throughout the elastic ranges of the specimens. Space limitations preclude the giving of all the data so a comparison will be made only on the basis of a nominal stress of 15,000 lb. per sq. in., for the purpose of showing the effects of the different factors on the strain readings, in the elastic range of the material. Because of the straight-line relationships previously noted, however, such a comparison would be valid for any other nominal stress within the elastic range.

RESULTS OBTAINED

In the tension tests of the rectangular sheet specimens, the strain gages were applied to both the edges and faces at the center of the reduced section. The results obtained for the nominal stress of 15,000 lb. per sq. in. are given in Table I. The strain readings at other stresses, however, had to be used to obtain the modulus of elasticity and yield-strength values given.

TABLE I.—SHOWING VARIATIONS IN TENSILE PROPERTIES OF SHEET SPECIMENS.

Thickness of Sheet, in.	Gage Line[a]	Young's Modulus of Elasticity, lb. per sq. in.	Variation in Modulus of Elasticity, per cent	Measured Stress, $\frac{P}{A} = 15{,}000$, lb. per sq. in.	Variation in Stress, per cent	Yield Strength, lb. per sq. in.	Variation in Yield Strength, per cent
$\frac{1}{4}$	No. 1	9 640 000	−3.35	15 690	+4.53	56 000	+0.04
$\frac{1}{4}$	No. 2	9 890 000	−0.89	15 340	+2.20	56 000	+0.04
$\frac{1}{4}$	No. 3	10 260 000	+2.89	14 280	−4.86	56 000	+0.04
$\frac{1}{4}$	No. 4	10 110 000	+1.38	14 725	−2.00	55 900	−0.13
$\frac{1}{8}$	No. 1	9 940 000	−0.90	15 470	+1.71	39 200	+0.26
$\frac{1}{8}$	No. 2	10 070 000	+0.40	15 440	+1.51	39 200	+0.26
$\frac{1}{8}$	No. 3	9 810 000	−2.20	15 650	+2.89	38 900	−0.51
$\frac{1}{8}$	No. 4	10 300 000	+2.70	14 285	−6.08	39 100	0
$\frac{1}{16}$	No. 1	9 800 000	−2.39	15 755	+4.92	39 000	−0.89
$\frac{1}{16}$	No. 2	9 930 000	−1.10	15 275	+1.73	39 400	+0.13
$\frac{1}{16}$	No. 3	10 040 000	0	14 900	−0.77	39 500	+0.38
$\frac{1}{16}$	No. 4	10 390 000	+3.49	14 130	−5.89	39 500	+0.38
$\frac{1}{64}$	No. 1	8 840 000	−9.78	15 870	+7.41	39 500	−1 06
$\frac{1}{64}$	No. 2	9 930 000	+1.36	14 325	−3.05	40 200	+0.69
$\frac{1}{64}$	No. 3	10 210 000	+4.22	14 280	−3.35	40 000	+0.19
$\frac{1}{64}$	No. 4	10 210 000	+4.22	14 620	−1.05	40 000	+0.19

[a] Gage lines Nos. 1 and 3 on edges and Nos. 2 and 4 on faces of specimens.

In thin specimens of this type appreciable variations in modulus determination would be anticipated. The magnitude of the variations found in these tests, however, would be somewhat decreased if the results from opposite sides of the specimen were averaged. The data given would appear to indicate that yield-strength determinations can be made with satisfactory results on either the edges or faces of the specimen.

The variations in strain, indicated in terms of stress, for the ½-in. diameter specimens, under the different conditions of testing used, are shown in Fig. 2. Some of the stress-strain curves for these specimens are shown in Figs. 3 and 4. It may be observed that under quite favorable conditions of testing wherein much care has been given to apparatus, preparation of specimens and testing procedure, measurable variations in strains will occur. It would seem apparent from the data that eccentric conditions of loading have a more pronounced effect on the strain readings than oblique loading conditions of similar magnitude. This is particularly so if the gage lines used happen to be in or near the plane of eccentricity. On the other hand, if the gage lines are in or near a plane normal to the plane of eccentricity quite satisfactory strain measurements may be obtained.

The marked differences in values of modulus shown by the individual gage line curves in Figs. 3 and 4 are very much minimized if not almost eliminated when values from opposite gage lines are averaged. This appears to be so even when the instruments are not at the center of the reduced length of the specimen. It is apparent from a consideration of the modulus values obtained on any three gage lines equally spaced around the specimen, that an average of any such three does not give as satisfactory results throughout, as an average of values from any two gage lines diametrically opposite.

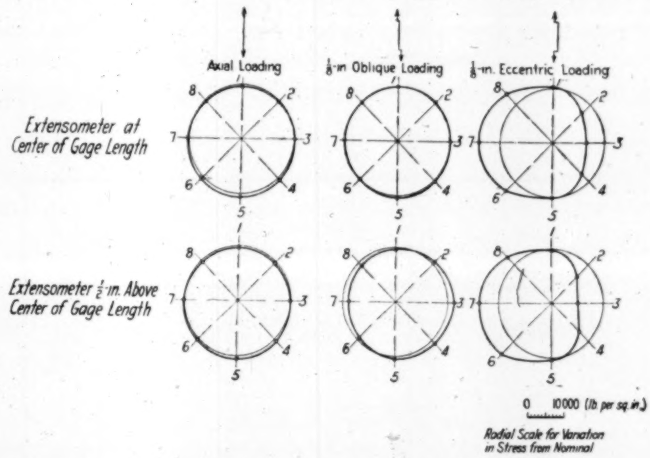

FIG. 2.—Variation of Stress from a Nominal Value of 15,000 lb. per sq. in. in Tension Tests of Standard $\frac{1}{2}$-in. Diameter A.S.T.M. Specimen.

The yield-strength values showed quite close agreement except in the case of the obliquely loaded specimens with the strain gages 0.5 in. above the center of the specimen.

The variations in strain, again in terms of stress, for a nominal stress of 15,000 lb. per sq. in., occurring in tubular tension specimens are shown in Fig. 5. With respect to the variables included, it would seem that there is not much difference in the strain readings obtained

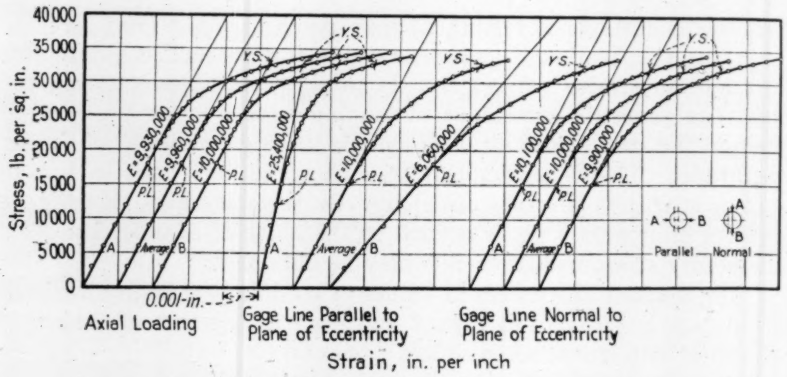

FIG. 3.—Stress-Strain Curves for Standard ½-in. Diameter Tension Specimen Under Axial and Eccentric Loads.

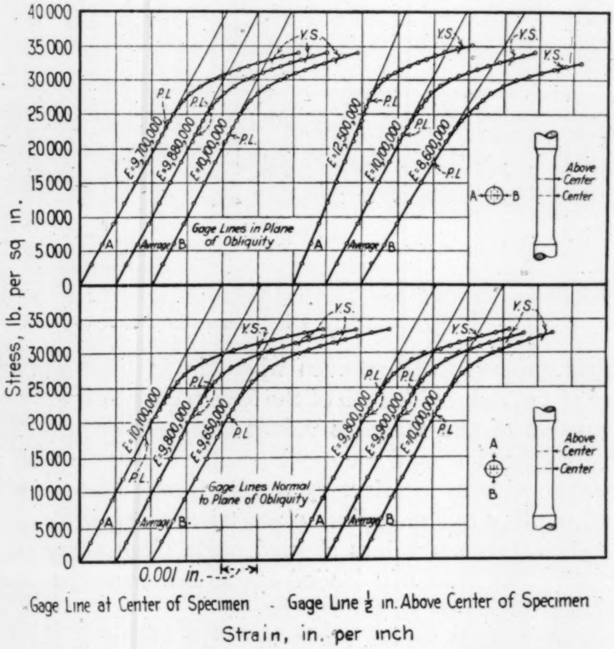

FIG. 4.—Stress-Strain Curves for a Tension Specimen Under Oblique Loading.

when 90-deg. and 120-deg. V-grips are used. In general, what differences there are would appear to be slightly in favor of the 120-deg. V-grips. The well fitting plugs used were machined so they had to be driven into the specimen, whereas the poorly fitting plugs had to be held in place until appreciable load was applied to the specimen. As would be expected appreciably more consistent readings were obtained with the better fitting plugs.

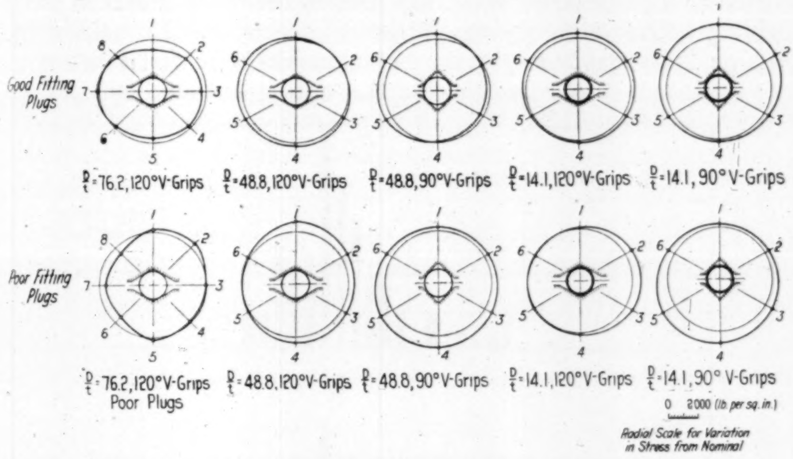
Good Fitting Plugs
Poor Fitting Plugs
D/t = 76.2, 120° V-Grips
D/t = 48.8, 120° V-Grips
D/t = 48.8, 90° V-Grips
D/t = 14.1, 120° V-Grips
D/t = 14.1, 90° V-Grips
D/t = 76.2, 120° V-Grips Poor Plugs
D/t = 48.8, 120° V-Grips
D/t = 48.8, 90° V-Grips
D/t = 14.1, 120° V-Grips
D/t = 14.1, 90° V-Grips
0 2000 (lb. per sq. in.)
Radial Scale for Variation in Stress from Nominal

FIG. 5.—Variation of Stress from a Nominal Value of 15,000 lb. per sq. in. in Tension Tests of Tubing.

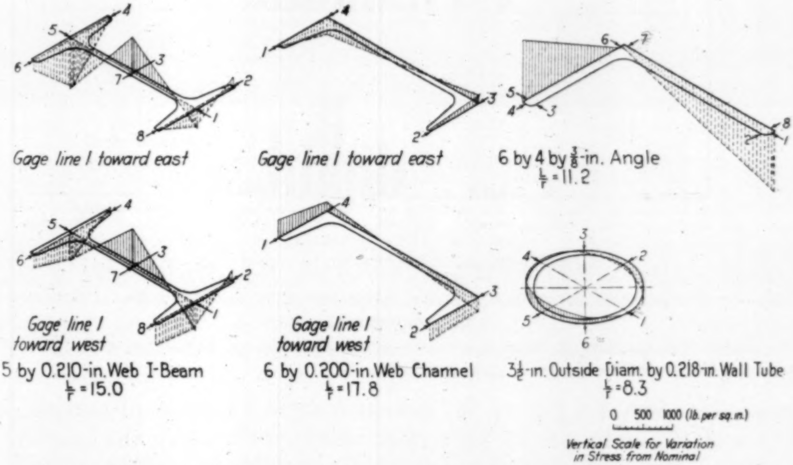
Gage line 1 toward east
Gage line 1 toward east
6 by 4 by 3/8-in. Angle
l/r = 11.2
Gage line 1 toward west
Gage line 1 toward west
5 by 0.210-in. Web I-Beam
l/r = 15.0
6 by 0.200-in. Web Channel
l/r = 17.8
3½-in. Outside Diam. by 0.218-in. Wall Tube
l/r = 8.3
0 500 1000 (lb. per sq. in.)
Vertical Scale for Variation in Stress from Nominal

FIG. 6.—Variation of Stress from a Nominal Value of 15,000 lb. per sq. in. Compression Tests of Various Shapes.

The variations in the measured strains, in terms of stress, for certain common structural shapes together with one tubular specimen, in compression are shown in Fig. 6. The variations in stress have been plotted to a much larger scale than used in the other figures in order to bring out more clearly the differences occurring. In making the tests on these specimens, strain measurements were first taken with the specimens in one position in the testing machine and a load applied in 3000 lb. per sq. in. increments until a nominal maximum stress of 18,000 lb. per sq. in. was attained; after which the load was removed, the specimens were rotated 180 deg. in the machine and the loading procedure repeated to the stress indicated, then carried beyond the yield strength and to failure. Certain differences in stress variations may be noted in the cases of the I-beam and channel specimens when they are rotated 180 deg. These differences may be attributed to slight errors in parallelism of the heads of the testing machine and

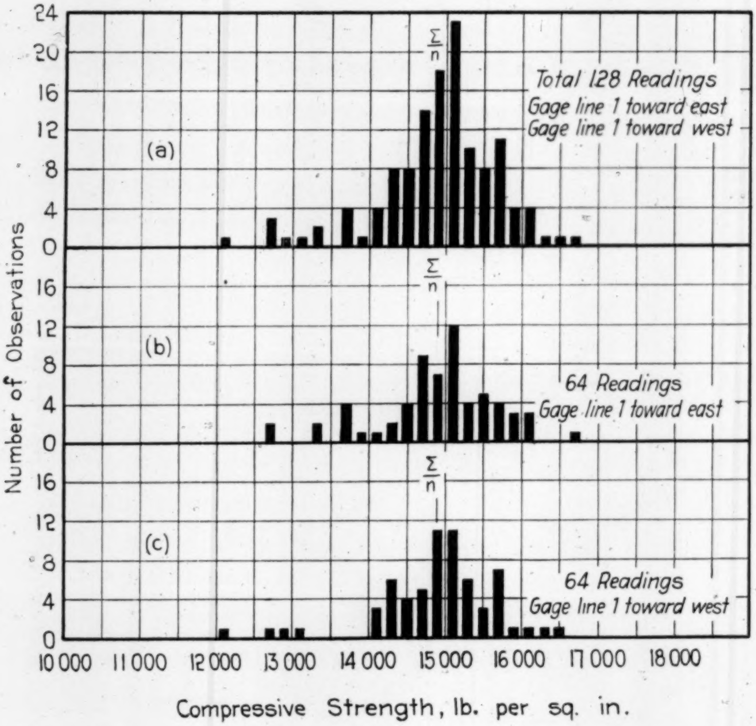

FIG. 7.—Frequency Diagrams for Strain Measurements on 5-in. I-Beam Specimen in Compression.

NOTE.—Each plotted point represents the average of eight readings, either eight tensometers on each gage line or each tensometer on eight gage lines.

slight errors in parallelism of the faces or ends of the specimens. In the case of the angle compression specimens, buckling of the longer outstanding leg took place early in the test with the result that the relief of strain in this portion of the specimen caused an increase in the strain in the other leg of the specimen. The values of stress indicated for this specimen are the averages of each set of three gage lines at the outer ends of the outstanding legs and two gage lines on the two faces near the corner of the angle. The variations in strain in the case of the tubular specimens are indeed of small magnitude.

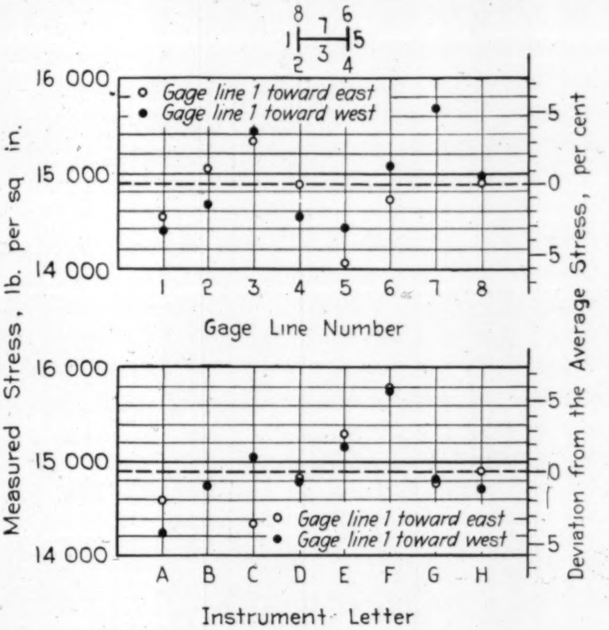

FIG. 8.—Chart Showing the Average Measured Stresses for Each Gage Line and Each Tensometer, for the 5-in. I-Beam in Compression.

In an attempt to give a better picture of the variations of strain measurements occurring in a compression specimen, the data for the I-beam specimen have been plotted in the form of frequency diagrams in Figs. 7 and 8. Using the methods advocated in the Society's Manual on Presentation of Data,[1] it is found that the measured average stress in this specimen is 14,890 lb. per sq. in., when the nominal value is 15,000 lb. per sq. in. Measured values ranging from 12,030 to 16,690 lb. per sq. in. are observed. Breaking down these data shown in Fig. 7 (*a*), so as to take account of the two positions

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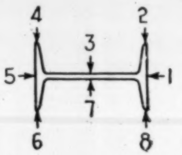
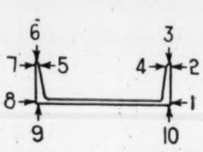
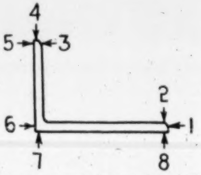
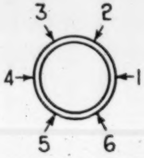
[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 451 (1933).

TABLE II.—VALUES OF YIELD STRENGTH AND MODULUS OF ELASTICITY FROM COMPRESSION TESTS ON STRUCTURAL SHAPES.









Gage Line	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Yield Strength, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Yield Strength, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Yield Strength, lb. per sq. in.	Variation from Average, per cent	Modulus of Elasticity, lb. per sq. in.	Variation from Average, per cent	Yield Strength, lb. per sq. in.	Variation from Average, per cent
	Gage Line 1 Toward East		Gage Line 1 Toward West				Gage Line 1 Toward East		Gage Line 1 Toward West											
1	10 600 000	+3.40	10 550 000	+3.43	41 000	+0.89	10 500 000	+0.96	9 900 000	−1.98	....	....	10 900 000	+6.85	32 600	0	10 300 000	+0.98	34 800	−0.29
2	10 250 000	0	10 200 000	0	40 700	+0.15	10 350 000	−0.48	9 800 000	−2.96	....	....	9 400 000	−7.84	32 000	−1.88	10 300 000	+0.98	34 900	0
3	10 000 000	−2.44	9 850 000	−3.43	40 500	−0.34	10 400 000	0	9 900 000	−1.98	52 500	0	10 200 000	0	[a]	....	10 200 000	0	35 200	+0.86
4	10 350 000	+0.98	10 400 000	+1.96	40 700	+0.15	10 400 000	0	10 150 000	+0.50	....	....	9 250 000	−9.30	32 600	0	10 200 000	0	34 600	−0.86
5	10 850 000	+5.85	10 600 000	+3.92	40 300	−0.84	10 100 000	−2.88	10 150 000	+0.50	....	....	9 200 000	−9.80	32 400	−0.62	10 100 000	−0.98	35 000	+0.29
6	10 450 000	+1.95	10 200 000	0	40 800	+0.39	10 400 000	0	10 200 000	+1.99	52 700	+0.38	10 000 000	−1.96	[a]	....	10 100 000	−0.98	35 000	+0.29
7	9 800 000	−4.40	9 800 000	−3.92	40 400	−0.59	10 400 000	0	10 600 000	+4.95	....	....	10 350 000	+1.47	[a]	....	....	....	....	....
8	10 300 000	+0.49	10 300 000	+0.98	40 700	+0.15	10 300 000	−0.96	10 450 000	+3.46	....	....	12 600 000	+23.50	[b]	....	....	....	....	....
9	....	....	....	....	....	....	10 300 000	−0.96	10 250 000	+1.48	52 100	−0.76	....	.....	....	....	....	....	....	....
10	....	....	....	....	....	....	10 300 000	−0.96	10 150 000	+0.50	52 000	−0.95	....	.....	....	....	....	....	....	....
Average	10 250 000		10 200 000		40 640		10 400 000		10 100 000		52 500		10 200 000		32 600		10 200 000		34 900	

[a] Stress-strain curve showed no departure from the straight line, that is, did not reach yield strength.

[b] Stress-strain curve showed a reversal in direction indicating a relief of stress after longer outstanding leg failed by buckling.

of the test specimen in the testing machine, we find about the same picture as shown in Figs. 7 (*b*) and (*c*). If the data, however, are plotted on the basis of gage line number and instrument letter, as shown in Fig. 8, and the results so obtained correlated with the information given in Fig. 7, we find that the low readings represent a combination of a low-reading instrument and a low-reading gage line, and in the same way the high values represent the combination of a high-reading instrument with a high-reading gage line. It is evident from a study of the diagrams in Fig. 8, that the errors resulting from location of gage lines are of the same order of magnitude as those caused by the instruments used. In general it will be observed that the instruments repeat quite satisfactorily with the exception of A and C. It so happens that these are two instruments which have been in service for a number of years, whereas the other instruments are new and have been used but a comparatively short time.

The modulus of elasticity and yield strength values from the compressive tests of structural-shape specimens are given in Table II. In the results obtained for modulus of elasticity, it will be noted that rather wide variations exist. In the case of the I-beam section, it may be further noted that the averaging of opposite gage lines, such as 1 and 5, 2 and 6, 3 and 7, or 4 and 8, does tend to decrease the amount of variation from the average value. The largest variation in modulus of elasticity value was obtained on the outer end of the longer leg of the unsymmetrical angle section indicating that bending of this portion of the specimen started at comparatively low loads.

Attention is called to the very satisfactory agreement of the yield strength values obtained on any gage line of the structural-shape specimens. The greatest observed variation is in the case of the unsymmetrical angle specimen.

CONCLUSIONS

Summarizing the results obtained in the investigation just described, it would seem evident that:

1. Considerable variations in strain and hence stress are to be anticipated in both tension and compression specimens, even under conditions of testing involving the use of careful technique and good apparatus.

2. The results obtained indicate definitely the necessity for uniform and axial loading of specimens, especially when determining the elastic modulus.

3. If such conditions could be attained, it would appear relatively unimportant whether strains are measured on one or more elements of the specimen.

4. Under conditions of some non-uniform or non-axial loading more satisfactory modulus values are obtained if strains on opposite elements of a specimen are averaged.

5. The test conditions for obtaining satisfactory yield strength values are much less exacting than those for obtaining satisfactory modulus values.

6. Strain readings obtained on any one element parallel to the axis of a specimen, either in the tension or compression test, will give quite satisfactory yield strength values.

These facts emphasize the necessity of considering the purpose of the test results in selecting suitable methods of testing.

Acknowledgment.—The author wishes to acknowledge the assistance of Messrs. M. Holt and J. O. Lyst in securing the test data given.

DISCUSSION

Mr. H. C. Mann[1] (*presented in written form*).—The conclusion drawn from Mr. Templin's experiments are for the most part in complete accordance with results obtained at the Watertown Arsenal Laboratory. In the testing of plate material $\frac{1}{4}$ in. and over in thickness, we have found it absolutely essential to use a type of extensometer which automatically reads the average of the strains, and have also obtained more satisfactory results when the strain gage is attached to the edges of the plate, rather than to the plate faces.

So far as modulus of elasticity is concerned, our experience has brought out that material condition as regards the presence of residual stress, is of far greater importance than the question of axial loading. The effects of non-axial loading are practically eliminated by the use of an extensometer which automatically reads the average of the strain on opposite elements of the specimen, but unless the material is completely relieved of residual stress, true modulus values cannot be obtained.

It is inferred from the statement in the last sentence under the section "Materials and Specimens" (page 184) that cold-worked steel would be expected to show similar stress-strain characteristics to high-strength wrought aluminum alloys. If this inference is intended we would point to the fact that steel which has been cold worked and subsequently stress relieved, and this is the only condition in which it should be tested, will exhibit the same stress-strain characteristics as before cold work, the only difference being in the higher proportional limit obtained. The true modulus value will remain the same regardless of the amount of cold work.

Mr. R. W. Vose[2] (*presented in written form*).—Mr. Templin's statement that "the average of three equally spaced gage lines (around a cylindrical tension specimen) does not give as satisfactory results as the average of two gage lines diametrically opposite" disturbed me since I have been using three equally spaced Huggenberger Tensometers in certain rather particular work. To clear up this point I have analyzed the general case and also Mr. Templin's data from several different points of view.

In the accompanying Fig. 1 are shown three different cases of stress distribution as they are ordinarily conceived according to the simple theory. The tension specimens are loaded with eccentricities of one-eighth, one-quarter, and one-half the radius of the specimen, respectively, and the resulting stresses shown to scale around the outside of the specimen. These stresses are also shown on the polar plot such as Mr. Templin uses in his Fig. 2 in which the stress at any point on the outside of the specimen is plotted to scale on the radius vector to that point.

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[1] Research Engineer, War Department, Watertown Arsenal, Watertown, Mass.

[2] Assistant in Materials Testing, Massachusetts Institute of Technology, Cambridge, Mass.

From the figures it may be readily seen that a perfectly regular stress distribution may be represented on a somewhat irregular polar plot, and that consequently this type of polar plot is not suited to simple visual analysis. A comparison with Mr. Templin's Fig. 2 will show that the stresses which he represents in this way are probably in very close agreement with the uniformly varying distribution of stress which the simple theory of eccentric loading calls for. For the case of uniformly varying stress over a circular section I would take it for granted that any number of equally spaced readings would average up correctly, but to make sure of this point I have obtained a rigorous mathematical proof which will not be given for lack of space.

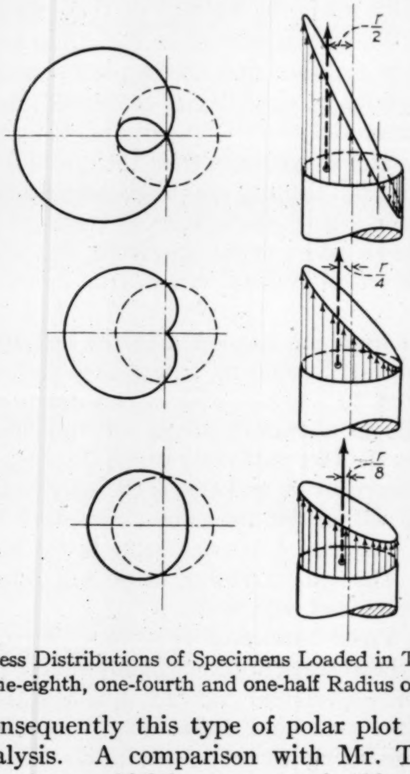

Fig. 1.—Showing Stress Distributions of Specimens Loaded in Tension with Eccentricities of one-eighth, one-fourth and one-half Radius of Specimen.

Since Mr. Templin plots eight equally spaced readings around his specimen, and makes no mention of having taken three equally spaced readings, I would infer that his conclusion regarding the latter arrangement have been drawn from the general appearance of the polar plot.

In view of the above I am led to the following conclusions:

1. The polar plot of stress, though correct, is misleading to visual inspection.

2. The stress on Mr. Templin's cylindrical specimens was uniformly varying within the usual limits of experimental determination.

3. The use of three equally spaced readings diminishes the probable error of the average over that given by two, and in addition gives a definite indication of the eccentricity, where two may not.

Another point of an entirely different nature may be noted on comparing my diagrams in Fig. 1 with Mr. Templin's Fig. 2. He states that his specimens were loaded with an eccentricity of $\frac{1}{8}$ in. or one-half the radius, which would correspond to the top diagram in Fig. 1. My diagram, based on the common theory and neglecting any local action of the fillets, etc., shows a very definite reversal of stress, and an actual loop on the polar plot, while Mr. Templin's experimental plot corresponds much more closely to my lower diagram with only $\frac{1}{32}$ in. eccentricity.

While I do not expect theory and practice to check exactly in this case, the difference should not be more than ten per cent or so, and this would indicate that the actual eccentricity in Mr. Templin's specimens was only a small fraction of what he intended. This situation may be easily explained if he loaded his specimens through screw end holders, since the slack in the screw threads might easily give $\frac{1}{8}$ in. and neutralize the intended offset. Accordingly I would like to inquire of Mr. Templin how he loaded his specimens and how he measured the eccentricity of the load.

Mr. Templin's Fig. 8 showing the percentage deviation of the various Huggenberger Tensometers checks very nicely with my own experiences. The largest variation is about six per cent while the average is two per cent, which shows that the various errors of calibration, mounting pressure, position, etc., are quite serious. It is only by a special optical calibration and a thorough investigation of the behavior of these instruments that I have been able to bring the errors within the one per cent that is desirable for research work.

Mr. Templin's conclusion that "considerable variations in strain and hence stress are to be anticipated in both tension and compression specimens, even under conditions of testing involving the use of careful technique and good apparatus" is only too true. It is practically impossible to obtain central loading with a 2-in. screw-end specimen with any of the so-called "swivel" holders, and furthermore a specimen once removed from the holders, or even slacked off, can never be re-loaded in the original manner. The most nearly perfect condition that I have discovered consists of loading a common 2-in. screw-end tension specimen in compression by means of ½-in. steel balls placed in the center holes. This is suitable only for work well within the elastic range, but it does give centering within about one per cent, and is certain to give conditions capable of being reproduced.

MR. L. B. TUCKERMAN[1] (*presented in written form*).—Anyone interested in accurate measurement of strain will find it profitable to study in detail the strain distributions which Mr. Templin shows. It is, for instance, obvious from his data that on round, solid or tubular specimens, two diametrically opposite gages will give a better measure of the average strain than three gages "at three equidistant points," which could also be predicted from theoretical considerations. If two gages are not adequate, then four should be used. That two gages are not always adequate for an accurate modulus determination can be seen from a study of Mr. Templin's Fig. 5. All of the curves, even with good fitting plugs, are definitely elliptical in shape, the ellipticity being more marked for the thinner walled (larger D/t) tubes.

We have found similar results at the National Bureau of Standards. A careful study was made some time ago on a chromium-molybdenum steel tube, diameter 2.502 in., average wall thickness 0.0343 in., $D/t = 72.9$, tested with a "wringing fit" mandrel in 120-deg. V grips. Eight (sometimes only four) optical strain gages were uniformly spaced around the tube at distances 3¾, 8, and 12 in. (middle of specimen) from the grips. In spite of care to secure axial loading, we found, as we had expected to find, definite indications of eccentricity of load. We were, however, surprised to see how little reliance could be placed on two diametrically opposite gages.

The average strain for the same load measured on two diametrically opposite gages 3¾ in. (1½ tube diameters) from the grips differed by over 19 per cent, depending upon the relative position of wedge grips and gages. When the distance from the grips was increased to 12 in. (middle of specimen) the difference between pairs of opposite gages decreased to less than 2.5 per cent. This approach to uniformity as the distance from the grips is increased is a striking illustration of St. Venant's "principle of the elastic equivalence of statically equipollent systems of load." In all cases, however, even when the gages were too near the grips for the local inequalities to disappear, the average of any set of four gages spaced 90 deg. apart agreed with the average of any other set of four spaced 90 deg. apart by less than $\frac{1}{3}$ per cent.

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

These results on thin-walled tubing, although their magnitude is larger than anticipated, are readily understood as consequences of the unsymmetrical gripping of the specimens. Not so clear are the reasons for the high measured strains in the web of the I-beams tested in compressions (see Fig. 6 of the paper). Were those perhaps caused by incipient buckling in the outstanding flanges, as in the case of the angle specimen?

His conclusions are an excellent summary of the data, but I find myself wondering about his last conclusion that "strain readings obtained on any one element parallel to the axis of a specimen, either in the tension or compression test, will give quite satisfactory yield strength values."

There is no question in my mind that this conclusion is justified for tests made on accurately dimensioned specimens in laboratories such as those under Mr. Templin's charge, where the testing fixtures are regularly inspected and adjusted, and only minor misalignments can exist. Testing equipment is not so carefully maintained in all testing laboratories and test specimens are not all accurately dimensioned. The average of two opposite gages will iron out, so far as yield strengths are concerned, fairly large eccentricities of load, and irregularities in specimens. Perhaps I should not question his conclusion but use it as an argument that the testing equipment in more testing laboratories should have as careful supervision.

MR. J. R. TOWNSEND.[1]—I should like to discuss briefly a point in the paper, namely, the accuracy of the tension test. On page 184 of the paper, it is stated that "The accuracy of each range used was well within ± 1 per cent." Referring to Table I of the paper, the $\frac{1}{4}$-in. thickness specimen which I take it was $\frac{1}{2}$ in. in width, had a measured stress of 15,000 lb. per sq. in.; the corresponding load is 1875 lb. If this load were measured on a 4000-lb. scale, one per cent error, taking the maximum error indicated, would be 40 lb. That corresponds to a two-per-cent error in the computed stress, or 320 lb. If the load were indicated on a 2000-lb. scale, the error then would be 20 lb. (or one per cent) and the stress 160 lb.

Keeping this in mind, it is mentioned in the paper that the error in the extensometer is one part in 120,000. This corresponds to a measurement in length or elongation of the specimen of 0.000008 in. and an error in strain of approximately 80 lb. for material with a modulus of elasticity of 10,000,000 lb. per sq. in. If the material had a modulus greater than 10,000,000 lb. per sq. in., for example steel where it is 30,000,000 lb. per sq. in., the error in measuring the strain due to the extensometer would be three times as great.

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[1] General Development Laboratory, Bell Telephone Laboratories, Inc., New York City.

Calibration data from a number of testing machines indicate that errors in the testing machine of the order of one-eighth to one-quarter of a per cent are possible when testing in the mid-range of the testing machine in determining the cord seals, and applying this to the figures previously given, it is seen that the error is in general less than the error in determining the strain. Extensometers read roughly one part in 100,000; accuracies greater than this are subject to some question. It is possible instead of using a 1-in. gage length, however, to use a larger gage length, and while this does not give a proportionate increase in accuracy, it certainly aids in readability, and in all probability gives a general over-all picture of the percentage elongation of the material.

In general it might well be stated that due to the developments in the testing machine and the accurate calibration that is possible with these machines at the present time, with the Moorehouse and other types of calibrating rings; the over-all accuracy of the testing machine exceeds the over-all accuracy of the extensometers that are available for stress-strain determinations. In ordinary routine testing, this is not of very great importance, but where accurate modulus determinations are necessary, and particularly in view of recent developments in the mechanical art, where it is necessary to know the modulus and to determine elastic hysteresis, it is very important that we have considerable improvement in the type and accuracy of extensometers. This discussion is offered, therefore, to call attention to the need for the development of more sensitive extensometers.

MR. R. L. TEMPLIN[1] (*author's closure*).—In regard to the discussion offered by Mr. Mann, the question of relative significance of initial stress in modulus of elasticity determinations might be somewhat controversial because the modulus may be determined on relatively small changes of stress which would not be materially affected by residual stress. An example of the determination of modulus at very low stresses is found in the work done by Messrs. Brombacher and Melton.[2] The suggested procedure for determining the modulus of elasticity of cold-worked materials by first annealing them is not I believe common practice in the testing of materials. Our own experience has been that the more satisfactory procedure is to first overload the cold-worked specimen above the yield value, then release the load and determine the modulus of elasticity by suitable stress-strain measurements. This procedure has been followed in some instances using as many as twenty overloadings with no further detectable change in modulus value after the first overloading.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] W. G. Brombacher and E. R. Melton, "Temperature Coefficient of the Modulus of Rigidity of Aircraft Instrument Diaphragm and Spring Materials," *Report No. 358*, Nat. Advisory Committee for Aeronautics (1930).

Mr. Vose refers to the statement that the average of any two opposite gage lines appears to give better results than the average value from three gage lines as interpolated from the curve shown. If the stress has a perfectly planar distribution across the section, the average of any two or more of gage lines equally spaced around the specimen will give the true average. If, however, the stress distribution is not truly planar, the more gage lines that are used uniformly distributed around the periphery, the more reliable the average value. Mr. Vose's use of three gage lines does not necessarily lead to a greater error than the use of two gage lines, but as pointed out in the paper, one pair of opposite gage lines spaced 180 deg. on solid specimens seemed to give better values than the results which would have been obtained from three gage lines.

In connection with the variations in stress caused by the eccentricities, it should be pointed out that while eccentricities were introduced in the loading of the specimens there was no assurance from the set-up that the intended eccentricity was actually obtained on the specimen because the loading shackles were relatively stiff compared to the specimen. It should be pointed out, however, that there was no intention of checking up on the actual eccentricities but they were introduced merely for the purpose of obtaining a variation in stress across the section.

In determining the yield strength of metals by means of a single gage line it is necessary, as Mr. Tuckerman has pointed out, that all testing equipment be frequently inspected and calibrated. Our procedure has been thoroughly to inspect, recondition and calibrate the testing machines in all of our laboratories at least once every six months.

One point Mr. Townsend made having to do with the accuracy of extensometers—I do not believe I specified that they were accurate to one part in 120,000, but were sensitive to that degree. There may be quite a difference between accuracy and sensitivity. The accuracy of the data given is more on the order of five per cent in some instances. High sensitivity without comparable accuracy may be very misleading and it should be emphasized, therefore, that extensometers with a high degree of both sensitivity and accuracy are desirable.

EFFECT OF SIZE AND SHAPE OF TEST SPECIMEN UPON THE OBSERVED PHYSICAL PROPERTIES OF STRUCTURAL STEEL

BY INGE LYSE[1] AND C. C. KEYSER[1]

SYNOPSIS

This paper presents the results of an investigation of the effect of size and shape of test specimens upon the observed Johnson's elastic limit, the tensile strength, the percentage of elongation in a 2-in. gage length, and the reduction of area of the fractured section. The specimens were machined from ¼-in., ½-in., and 1-in. thick plates which were rolled from the same heat of structural grade steel. The size and shape of the test specimens varied from ¼-in. to 1-in. diameter bars and from ¼ by ¼-in. to 1 by 4-in. rectangular sections.

The results showed that the size and shape of the test specimens had practically no effect upon either the Johnson's limit or the tensile strength. The observed percentage elongation in 2 in. increased and the reduction of area decreased with the increase in ratio of width to thickness of the rectangular specimens. For round bars the percentage elongation increased with the increase in diameter while the reduction in area remained practically constant. In general, the results of this investigation agreed well with previous data available on this subject.

INTRODUCTION

The question of how the size and shape of the test specimen influence the secured test results is frequently raised in connection with tests of steel and other materials. Recent investigations have shown that the strength of a large concrete specimen is considerably less than that of a small one,[2] a 36 by 72-in. cylinder showing only from 80 to 85 per cent of the strength of a 6 by 12-in. cylinder. Investigations on steel have shown only slight variations in strength due to the size and shape of the test specimens, but an uncertainty of the amount of these variations has been general among manufacturers and users of structural steel. The effect of the thickness of the rolled steel section has also been questioned; experiences having shown that a thick section often has a lower yield point than a thin section of the same material. For the purpose of securing more data on these questions than already available in engineering literature, an investigation was carried out at Lehigh University, the Bethlehem Steel Co. furnishing the material which was a product of the Sparrows Point Plant. In addition to the Johnson's limit and tensile strength of the specimens, observations were also made on the elongation in 2 in. and the reduction of area of the fractured section.

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[1] Research Associate Professor of Engineering Materials, and Laboratory Assistant in Civil Engineering, respectively, Lehigh University, Bethlehem, Pa.

[2] Arthur Ruettgers, "Mass Concrete as Affected by Size of Aggregate and Related Factors," *Proceedings*, Am. Concrete Inst., Vol. 30, p. 27 (1934).

Special acknowledgment is due Mr. V. E. Ellstrom, Manager of Sales Engineering, Bethlehem Steel Co., for securing the steel for the investigation.

EARLIER STUDIES BY BACH AND MOORE

A summary of Bach's experimental results on the effect of size and shape of specimens is given in his textbook.[1] For the effect of the diameter of the test bar on strength, elongation, and reduction of area, he concluded that:

1. The strength decreased slightly with the increase in diameter of the specimens.
2. The elongation was independent of the diameter and gage length as long as the ratio between gage length and diameter remained constant.
3. The reduction in area was independent of the diameter of the specimens.

Experiments on circular, rectangular, and H-sections of nearly equal cross-section showed that the circular section gave the highest yield point value, the rectangular section gave an intermediate, and the H-section the lowest value. The same relationship also existed for the tensile strength, although to a lesser degree.

On the whole, Professor Bach's investigations indicated that the size and shape of specimens had sufficient influence on the results to warrant further experimental verifications.

Professor H. F. Moore[2] summarizes much of the data available on the problem of effect of size and shape of test specimens of steel and also presents data not previously published. The most important conclusions of this study were "that there is no great difference in results between tests made on round specimens and tests made on flat specimens whose width is not more than four times the thickness."

"In general the test results showed no very marked advantage of one type of specimen over another, or any systematic difference in test results due to difference in ends or methods of gripping."

"All the test data indicated that the reduction of area at fracture is very slightly affected by the value of $\sqrt{a'}/l$ for a specimen, and that such variations as are shown are sometimes in one direction, and sometimes in the other."

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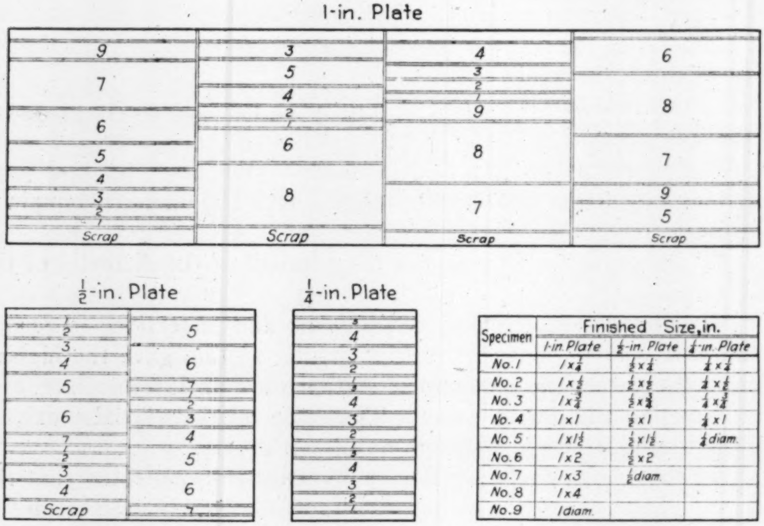
[1] C. Bach, "Elastizität und Festigkeit," Fifth Edition, Julius Springer, Berlin (1905).

[2] H. F. Moore, "Tension Tests of Steel with Test Specimens of Various Size and Form," *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part I, p. 403 (1918).

"All the test data show that the effect of variation of $\sqrt{a'}/l$ on the percentage of elongation is very pronounced."

OUTLINE OF TEST

In order that the study be limited to the effect of size and shape of test specimens only upon the observed results, the steel plates used were taken from the same heat. The chemical analysis of the steel was reported as 0.15 per cent carbon, 0.48 manganese, 0.014 phosphorus, and 0.028 sulfur. Three thicknesses of plate were used, namely, 1 in., $\frac{1}{2}$ in., and $\frac{1}{4}$ in. All the plates were 15 in. wide with sheared edges. The test specimens were machined from these plates in accordance with the plan shown in Fig. 1. The specimens varied in size from $\frac{1}{4}$ by $\frac{1}{4}$ in. to 1 by 4 in. Bars with center portion turned down to $\frac{1}{4}$-in., $\frac{1}{2}$-in., and 1-in. diameter were also included. Four inches of the center portion of all specimens were machined to the correct size. The elongation was measured on the 2-in. portion at the point of failure. Three specimens of each kind were used, making a total of 114 specimens for the 38 different conditions included in this investigation. A tabulation of the different specimens machined from each plate is shown in Fig. 1. For the $\frac{1}{4}$-in. plate all specimens



Specimen	Finished Size, in.		
	1-in. Plate	½-in. Plate	¼-in. Plate
No. 1	1×¼	½×¼	¼×¼
No. 2	1×½	½×½	¼×½
No. 3	1×¾	½×¾	¼×¾
No. 4	1×1	½×1	¼×1
No. 5	1×1½	½×1½	¼ diam.
No. 6	1×2	½×2	
No. 7	1×3	½ diam.	
No. 8	1×4		
No. 9	1 diam.		

FIG. 1.—Lay-Out of Specimens from 1, $\frac{1}{2}$ and $\frac{1}{4}$-in. Plates.

Table I.—Results on ¼-in. Thick Specimens.

Each value is an average of three specimens.

Size of Specimen, in.	Johnson's Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Ratio, Johnson's Limit to Tensile Strength, per cent	Elongation in 2 in., per cent	Reduction of Area, per cent	Modulus of Elasticity, lb. per sq. in.
¼-in. Thickness, As Rolled						
¼ diam	42 700	60 510	70.6	34	67	29 600 000
¼ by ¼	39 840	58 960	67.7	31	65	29 545 000
¼ by ½	40 160	59 360	67.9	39	61	29 565 000
¼ by ¾	39 400	59 090	66.7	43	60	29 560 000
¼ by 1	39 210	59 510	65.9	45	56	29 310 000
Average	40 260	59 490	67.8	38	62	29 510 000
¼-in. Thickness Cut from Rolled ½-in. Plate						
¼ diam	36 730	59 470	61.7	31	67	29 435 000
¼ by ¼	37 040	60 350	61.4	34	64	29 555 000
¼ by ½	37 680	60 570	62.2	40	61	30 090 000
¼ by ¾	36 980	60 910	60.7	42	59	29 755 000
¼ by 1	37 090	61 160	60.6	44	57	30 065 000
Average	37 100	60 490	61.3	38	62	29 790 000
¼-in. Thickness Cut from Rolled 1-in. Plate						
¼ diam	31 280	59 760	52.4	30	65	28 475 000
¼ by ¼	25 250	58 850	43.0	36	63	29 265 000
¼ by ½	26 020	58 250	44.7	39	62	29 530 000
¼ by ¾	26 000	58 160	44.7	41	57	29 700 000
¼ by 1	27 280	58 380	46.7	47	55	28 785 000
Average	27 160	58 680	46.3	39	60	29 155 000
Average for All ¼-in. Specimens						
¼ diam	36 890	59 910	61.6	32	66	29 180 000
¼ by ¼	34 050	59 390	57.4	34	64	29 450 000
¼ by ½	34 620	59 390	58.3	39	61	29 730 000
¼ by ¾	34 130	59 390	57.4	42	59	29 670 000
¼ by 1	34 530	59 680	57.7	45	56	29 390 000
½-in. Thickness As Rolled						
½ diam	37 610	60 030	62.7	40	68	29 570 000
½ by ¼	38 480	60 240	63.9	39	62	29 615 000
½ by ½	36 920	59 680	61.9	42	64	30 045 000
½ by ¾	36 930	59 700	61.8	48	64	29 800 000
½ by 1	36 760	59 780	61.5	50	61	30 155 000
½ by 1½	36 810	60 020	61.3	52	59	29 585 000
½ by 2	36 310	59 790	60.7	56	56	29 295 000
Average	37 120	59 890	62.0	47	62	29 720 000
½-in. Thickness Cut from Rolled 1-in. Plate						
½ diam	29 900	58 290	51.2	43	65	29 330 000
½ by ¼	27 830	58 700	47.4	37	59	28 685 000
½ by ½	27 650	58 050	47.6	41	61	29 300 000
½ by ¾	26 430	58 190	45.4	45	59	29 085 000
½ by 1	26 750	58 570	45.6	46	57	30 055 000
½ by 1½	25 500	58 510	43.5	49	55	29 600 000
½ by 2	28 080	57 890	48.5	53	52	28 690 000
Average	27 450	58 310	47.0	45	58	29 245 000
Average for All ½-in. Specimens						
½ diam	33 760	59 160	57.0	42	67	29 450 000
½ by ¼	33 160	59 470	55.7	38	61	29 150 000
½ by ½	32 290	58 870	54.8	42	63	29 670 000
½ by ¾	31 680	58 950	53.7	47	62	29 440 000
½ by 1	31 760	59 180	53.6	48	59	30 100 000
½ by 1½	31 160	59 270	52.5	51	57	29 590 000
½ by 2	32 200	58 840	54.6	55	54	28 990 000
1-in. Thickness As Rolled						
1 diam	30 570	58 440	52.3	51	62	28 470 000
1 by ¼	30 420	59 190	51.3	42	54	29 150 000
1 by ½	31 920	59 010	54.0	46	60	28 950 000
1 by ¾	31 580	58 960	53.6	51	61	29 715 000
1 by 1	31 500	58 100	54.2	55	62	29 600 000
1 by 1½	30 620	59 020	51.8	57	59	30 040 000
1 by 2	31 430	58 910	53.4	62	58	29 465 000
1 by 3	32 300	58 580	55.1	67	54	28 860 000
1 by 4	31 800	56 770	56.0	71	50	28 715 000
Average	31 350	58 550	53.5	56	58	29 220 000

had the full thickness of the plate. For the ½-in. plate one group of specimens had the full thickness of the plate, while another group was machined down to the ¼-in. center portion of the plate. For the 1-in. plate, full thicknesses, ½-in. center portion and ¼-in. center portion were used.

All specimens were tested in screw-power machines at a speed of about 0.05 in. per min. up to the total load. Huggenberger Tensometers with 1-in. gage length were used for deformation observations on all of the specimens. These instruments remained on the specimens until an appreciable plastic flow had taken place. Since no drop of the beam took place on any of the specimens from the 1-in. plate, the Johnson's limit,[1] instead of the yield point, was used as the basis for comparison.

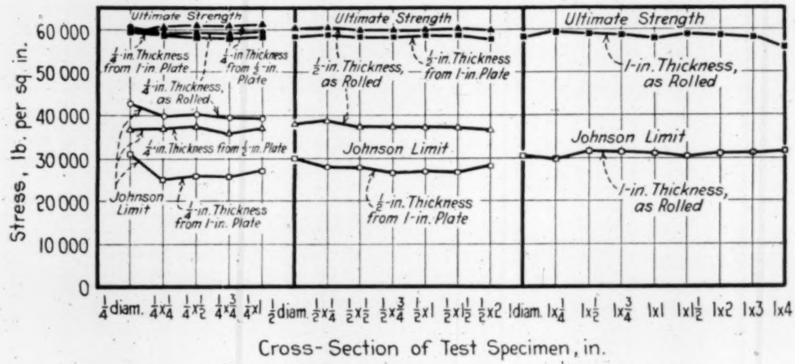


FIG. 2.—Effect of Size and Shape of Test Specimen on Strength Results.

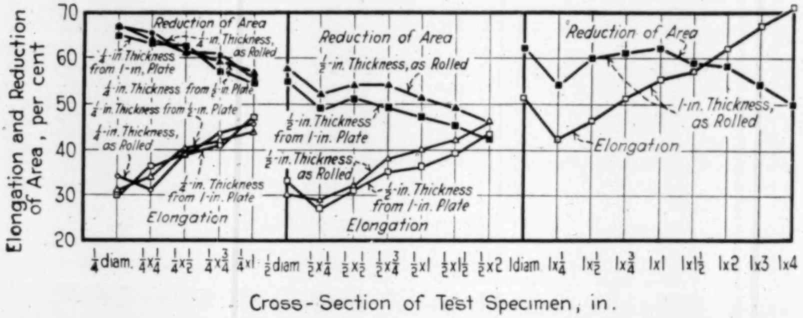


FIG. 3.—Effect of Size and Shape of Test Specimen on Elongation and Reduction of Area.

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[1] The stress at which the rate of deformation is 50 per cent greater than the initial rate of deformation.

TEST DATA

The average results of the three specimens for each condition are presented in Table I. A study of the effect of the size and shape of test specimen on the Johnson's limit and the tensile strength is given in Fig. 2 for each of the three $\frac{1}{4}$-in. plates, the two $\frac{1}{2}$-in. plates and the one 1-in. plate. The corresponding studies of elongation and reduction in area are presented in Fig. 3. The effect of the ratio between the two sides of the rectangular specimens both for Johnson's limit and tensile strength was also plotted and found to be practically negligible. Its effect upon the elongation and reduction of area is shown in Fig. 4. The effect of the thickness of the plate on Johnson's limit and tensile strength and on the elongation and reduction of area is given in Fig. 5.

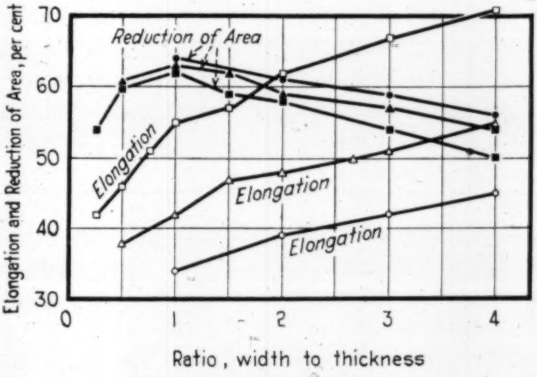

FIG. 4.—Effect of Width-Thickness Ratio of Test Specimen on Elongation and Reduction of Area for Plates of Various Thicknesses.

DISCUSSION OF RESULTS

A very high degree of uniformity was obtained in all of the specimens tested. Figure 2 shows that for rectangular cross-sections varying from $\frac{1}{4}$ by $\frac{1}{4}$ in. to $\frac{1}{4}$ by 1 in. there was no consistent difference either in the Johnson's limit or the tensile strength. However, the circular $\frac{1}{4}$-in. bar showed a slightly higher Johnson's limit than did the $\frac{1}{4}$-in. rectangular sections taken from the $\frac{1}{4}$ and 1-in. plates. Neither the $\frac{1}{2}$-in. nor the 1-in. diameter bars gave results appreciably different from the rectangular sections. For the $\frac{1}{2}$-in. and 1-in. thick material, all specimens from each thickness gave very nearly the same Johnson's limit and tensile strength. However, there is considerable difference for the Johnson's limit for the different plates as rolled, as seen from Table I. The tensile strength showed only a very slight variation between plates of different thickness as rolled. The shape of the test specimen did not affect either the Johnson limit or the tensile strength, although the ratio between the width and the thickness of the specimens varied from 0.25 to 4.0.

The elongation in 2 in. and in the reduction of area were affected appreciably by the size of the test specimen as shown in Fig. 3. The elongation for the $\frac{1}{4}$-in. plates varied from an average of about 32 per cent for a $\frac{1}{4}$-in. diameter round specimen to about 45 per cent for a $\frac{1}{4}$ by 1-in. rectangular specimen, the increase being uniform with the increase in size of specimen. For the $\frac{1}{2}$-in. thickness the elongation increased from about 38 per cent for a $\frac{1}{2}$ by $\frac{1}{4}$-in. specimen to about 55 per cent for a $\frac{1}{2}$ by 2-in. specimen. For the 1-in. plate the elongation varied from 42 per cent for a 1 by $\frac{1}{4}$-in. specimen to 71 per cent for a 1 by 4-in. specimen. Figure 4 shows how the average elongation

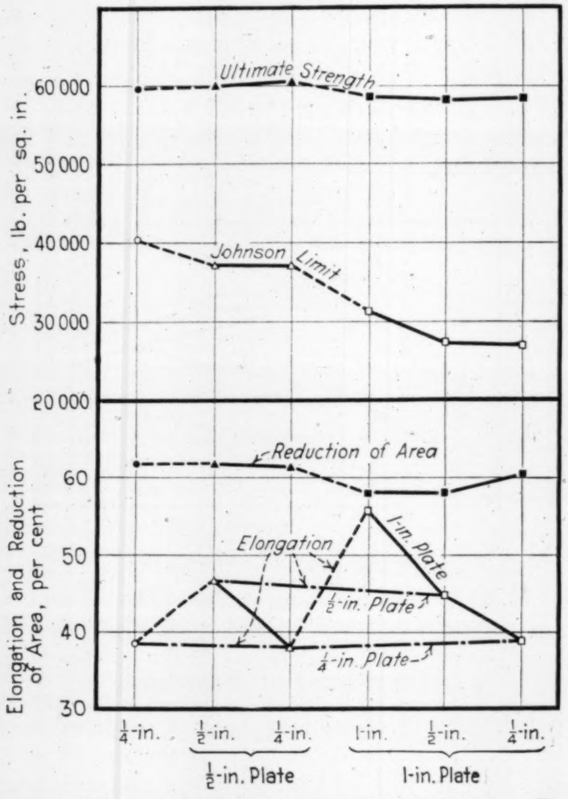

FIG. 5.—Effect of Thickness of Plate on Strength Results and Elongation and Reduction of Area.

in 2 in. increased with the increase in ratio between width and thickness of the specimens. It should be kept in mind that the ratio between gage length and cross-sectional area varied greatly for these specimens. Figure 4 also shows that the elongation was larger for the ½-in. material than for the ¼-in. material and larger for the 1-in. plate than for the ½-in. thickness. This is due to the fact that each cross-sectional dimension of the specimen of a given width-thickness ratio was twice as large for the ½-in. as for the ¼-in. material, and twice as large for the 1-in. as for the ½-in. material. As shown in Fig. 4, the elongation in 2 in. for the three thicknesses of plates as rolled was nearly the same when observed on the same size test specimen. Consequently the variation in elongation shown in Fig. 4 was due almost entirely to the difference in size of the specimens used.

The reduction of area showed a uniform decrease with the increase in ratio between the sides of the rectangular sections and also with increase in size of the test specimen. Figure 3 shows that the reduction of area for ¼-in. material decreased from about 66 per cent for the ¼-in. diameter specimen to about 56 per cent for the ¼ by 1-in. specimen. For the ½-in. material this decrease was from about 67 per cent for the ½-in. diameter specimen to about 54 per cent for the ½ by 2-in. specimen, and for the 1-in. plate from 62 per cent for the 1-in. diameter specimen to 50 per cent for the 1 by 4-in. specimen. From Fig. 4 it is noted that when the ratio between the width and thickness increased from ¼ to 1 there was an increase in the reduction of area, and when the ratio increased from 1 to 4 there was a uniform decrease in reduction of area. Thus the square section gave the greatest reduction of area for rectangular cross-sections. In general the circular specimens had reductions of area slightly greater than those for square sections.

An illustration of the effect of the thickness of the rolled plate upon the strength results is shown in Fig. 5. Johnson's limit showed a marked decrease with increase in thickness of the plate, due principally to the amount of rolling on the sections as a whole, rather than to the effect of the rolled surface. For the ½-in. plate, the ¼-in. center portion had just about the same Johnson's limit as did the full section. For the 1-in. plate, however, the ½-in. and ¼-in. center portions were about equal, but both were somewhat lower than was the full section. The tensile strength was only slightly affected by the thickness of the plate.

The effect of the thickness of the rolled plate on the elongation and reduction in area is also shown. It is noted that for the same size test specimens there was no appreciable variation in the elongation of the three plates. However, as previously pointed out, the size of the test specimen had a marked effect upon the elongation in 2 in.

SUMMARY

The most important results of this investigation may be summarized as follows:

1. The size and shape of the test specimen had practically no effect upon the observed Johnson's limit or the tensile strength of the material.
2. The elongation in 2 in. increased with the size of the test specimen.
3. The reduction of area of the fractured section decreased with increase in the width-thickness ratio of the rectangular sections, but remained nearly constant for circular bars of different diameters.
4. The ratio between Johnson's limit and tensile strength was nearly constant for the different sizes and shapes of test specimens, but showed a marked difference for different thickness of plate as rolled.
5. The thickness of the plate as rolled had a marked effect upon the Johnson limit, but only slight effect upon the tensile strength.
6. Only slight differences in results were obtained on specimens having the full thickness of the rolled plates and on specimens machined from the center portion of the plate.
7. The results of this investigation substantiate those of previous studies which have shown that the observed strength properties are little, if any, affected by the size and shape of the test specimen, while both elongation in 2 in. and reduction of area are affected appreciably.

DISCUSSION

Mr. R. L. Templin[1] (*presented in written form*).—We are indebted to the authors of this paper for giving us additional data showing the effects of size and shape of test specimen on the mechanical properties of structural steel. As they have pointed out, their results substantiate those of previous similar studies, notably those by Bach and Moore. It would appear that the results they have obtained might further be correlated with those presented before this society by Nichols, Taylerson and Whetzel[2] and the writer.[3] Likewise there have been similar studies by a number of foreign investigators, notably Bertella.[4]

The relationship between elongation on a fixed gage length, say 2 in., and the total cross-sectional area of the specimen for some metals is shown in the accompanying Fig. 1. This relationship may be expressed by a modified form of Bertella's equation as follows:

$$e = KA^n$$

where e = per cent of elongation in 2 in.,
A = the cross-sectional area of the reduced portion of the specimen, and
K and n = constants.

The data so far available would indicate that the value of the constant n is in the neighborhood of 0.1 for properly annealed wrought metal. The value of this constant increases with strain hardening or cold working, as may be seen by comparing the curves for annealed and hard-rolled pure aluminum.

The results given by the authors of this paper have also been plotted in Fig. 1 in comparison with those for mild steel obtained by Gordon and Beare.[5] The value of n obtained from their results is somewhat greater than 1, suggesting that the structural steel which they used in their experiment possessed a small amount of strain hardening.

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] J. T. Nichols, E. S. Taylerson and J. C. Whetzel, "Tension Test Specimens for Sheet Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 259 (1927).

[3] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 378 (1926).

[4] C. A. Bertella, "Sulle Prove de Tensione dei Metalli," *Giornale del Genio Civile*, Vol. 60, June, 1922, p. 343.

[5] William Gordon, "Sur la Variation Apparenti de la Ductilite Suivant la Largeur de l'Eprouvette," *Revue de Métallurgie*, October, 1925.

T. Hudson Beare and William Gordon, "The Influence of the Width of the Specimen upon the Results of Tensile Tests of Mild Steel and Rolled Copper," *Engineering* (London), September 9, 1921.

MR. R. L. KENYON.[1]—Supplementing Mr. Templin's remarks, I wish to point out that the data presented by the authors can be interpreted in a slightly different way by computing the so-called "slenderness ratio" for the various shaped specimens. This is determined by dividing the gage length by the square root of the cross-sectional area. The percentage of elongation has been found[2] to be practically the same for tension test specimens of different proportions providing the slenderness ratio is constant. This was shown particularly well in some tests reported by Mr. R. L. Templin.[3] In view of these considerations it would seem advisable to consider the relationship between the slenderness ratios and the elongation values given in this paper.

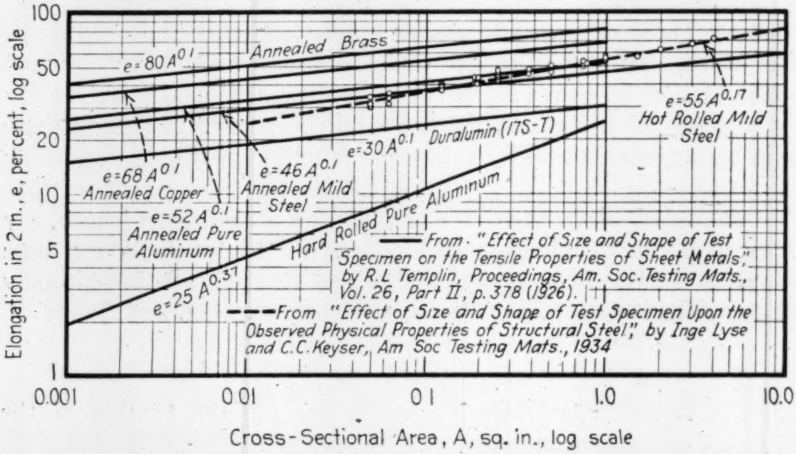

FIG. 1.—Relation Between Elongation and Cross-Sectional Area for Some Metals.

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[1] Supervising Research Engineer, The American Rolling Mill Co., Middletown, Ohio.

[2] M. J. Barba, "Résistance des materiaux," *Memoires et Compte Rendus des Traveaux*, Société des Ingenieurs Civils, Part I, p. 682 (1880).

G. F. Swain, "Structural Engineering, Strength of Materials," p. 68, McGraw-Hill Book Co., Inc., New York City (1924).

R. G. Batson and J. H. Hyde, "Mechanical Testing," p. 116, E. P. Dutton and Co., New York City (1922).

Ewing, "Strength of Materials," p. 47, Cambridge University Press, London (1906).

S. Timoshenko and J. M. Lessels, "Applied Elasticity," p. 362, Westinghouse Night School Press, East Pittsburgh, Pa. (1925).

J. B. Johnson, "Materials of Construction," Sixth Edition, p. 106, John Wiley and Sons, Inc., New York City (1926).

R. L. Templin, "Methods for Determining the Tensile Properties of Thin Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 235 (1927).

J. T. Nichols, E. S. Taylerson and J. C. Whetzel, "Tension Test Specimens for Sheet Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 259 (1927).

[3] R. L. Templin, "Effects of Size and Shape of Test Specimen on the Tensile Properties of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 378 (1926).

In order to show that this relationship is more significant than that of elongation *versus* ratio of width to thickness, which the authors have done in their Fig. 4, I have tabulated the slenderness ratios and elongation values for several specimens reported in Table I of the paper:

DIMENSIONS OF SPECIMEN, IN.	RATIO OF WIDTH TO THICKNESS, w/t	SLENDERNESS RATIO, $L/\sqrt{A}$	ELONGATION IN 2 IN., PER CENT
$\frac{1}{2}$ by $\frac{1}{2}$	1	4.00	42
1 by $\frac{1}{4}$	$\frac{1}{4}$	4.00	42
$\frac{1}{2}$ by $1\frac{1}{2}$	3	2.31	51
1 by $\frac{3}{4}$	$\frac{3}{4}$	2.31	51
$\frac{1}{2}$ by 2	4	2.00	55
1 by 1	1	2.00	55

These computations show that the elongation bears no relation to the proportions of the test specimen, but does vary with the value of $L/\sqrt{A}$. The data check the latter relationship so well that it must be concluded that the authors have conducted their tests with considerable care as to uniformity of material and preparation of specimens.

MR. I. F. MORRISON[1] (*by letter*).—In studies of this kind, where the influence of one factor is sought, all other factors should be maintained constant, or as nearly so as possible, or their influence should be taken into account and eliminated in the process of interpretation of the results. Unfortunately, there is no indication in the paper that complete precaution was taken against disturbing influences. The material in question was hot-rolled steel of low-carbon content. The chemical analysis given is presumably the ladle analysis. The writer has found by experience that this is not always accurate enough where careful investigations are in hand. In addition, there is no information given as regards grain size of the material for each of the three thicknesses of plate and for the bars. It seems to the writer that this is a highly important matter for although grain size may have little effect on the ultimate strength—within limits of course—it can have a very appreciable effect on the Johnson's limit. This is a possible explanation of the rather wide range of values for the Johnson's limit for the various plate thicknesses. The thinner plates are apt to be finished at a lower temperature, which would, if the temperatures were all above the critical range, tend to leave that material with a finer grain structure which in turn would give a higher Johnson's limit. It is to be regretted that photomicrographs of the material are not included in the paper.

Furthermore, if, before the finishing pass through the rolls, the temperature has dropped into or below the critical range the material will likely possess internal stresses which, especially at low loads, influence very markedly the extensometer readings and affect the determination of the modulus of elasticity. Machining may produce some internal stress and nearly always leaves badly distorted grains near the surface. Such distorted and even ruptured grains are especially apt to be present at the corners of rectangular specimens. These can be removed by care and the production of a fine surface finish. Preferably, specimens should be carefully annealed. There is nothing in the paper on the surface finish or heat treatment of the specimens. Nor do the authors indicate how they have dealt with these matters.

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[1] **Professor of Applied Mechanics, University of Alberta, Edmonton, Alta., Canada.**

It would be interesting to know also of the location of the extensometers on the rectangular pieces, that is, were they on the edge or on the wide face or on both? Furthermore, additional information concerning the depth of shoulder and the shape of the transition from the end portion and the central portion would seem desirable, especially for the larger specimens. These items are capable of producing a difference in the observed modulus of elasticity.

From Fig. 4 of the paper it is quite evident that the elongation in 2 in. increases with the width-thickness ratio, and with the size of the specimen. This conclusion, however, is to be expected because in each case for the wider and the larger specimen there is a larger volume involved in the plastic deformation of the material. These results are in accord with Barba's law of similarity, which means that the gage-length should be varied with the square root of the cross-sectional area. The writer feels that these tests would have been of greater value had this law of similarity been observed. There could then have been a direct comparison with Barba's work and the effect of width apart from size could have been observed.

The most general conclusion one can draw from these tests concerning the elongation is that it is meaningless unless determined from a standard specimen and that the results are of comparative value only. There is, strictly speaking, no intrinsic physical property of the material itself expressed by the "elongation." It is evident that the round specimen is to be preferred as a standard.

In regard to the reduction of area, the results are again in accord with what would be expected of the influence of shape on it. After necking starts, a three-dimensional stress condition arises. With circular sections, the lateral stresses are theoretically equal, whereas with rectangular sections they are not. For this reason, the shearing stresses on planes at 45 deg. to the specimen axis are unequal and, furthermore, as necking develops, the areas over which these shearing stresses are distributed are no longer equal. From this it is easy to see that the total plastic deformation will differ with the shape of the cross-section as indicated by the results of the tests.

In the reduction of area graph, Fig. 4 of the paper, the points at width-thickness ratio $\frac{1}{4}$ should agree with that at 4 and at $\frac{1}{2}$ with that at 2. The agreement is evident from the figure. There is thus no effect of size of specimen present as suggested in conclusion 7 of the paper. On the other hand, the effect of size is shown on the elongation graphs.

It is apparent from these results that with a larger width-thickness ratio, plastic flow is not permitted to take place for as long a time as for a smaller ratio. In other words, the tear resistance is overcome sooner for a larger width-thickness, thus putting an earlier end to the slip-flow process which results in a smaller reduction of area.

MESSRS. INGE LYSE[1] AND C. C. KEYSER[1] (*authors' closure by letter*).—The authors appreciate very much the additional information regarding the effect of size and shape of test specimens contributed by the discussions of Messrs. Templin and Kenyon. Particularly valuable are the data contained in Fig. 1 of Mr. Templin's discussion, as this diagram gives an excellent illustration of the behavior of widely different materials. Mr. Kenyon has illustrated in tabular form what was illustrated graphically in Figs. 4 and 5, namely, that the size or area of the specimen was important for the measured elongation in 2 in.

Unfortunately, the authors were limited to the control possible in present-day steel manufacturing practice and therefore could not eliminate those disturbing influences mentioned by Mr. Morrison. The three steel plates used in the investigation came from the same heat and the given analysis represents this heat. The machining of the test specimens was done very carefully. No annealing or special surface treatment was given. The extensometers were located at the centers of the narrow faces in all rectangular specimens and all specimens had a $\frac{1}{4}$-in. shoulder at the end of the machined central portion. Unfortunately the laboratory had no apparatus available for taking photomicrographs of the material in the plates used. Messrs. Templin and Kenyon answer very well the question concerning the elongation in 2 in. for specimens of various shapes. Mr. Morrison correctly points out that there is no appreciable effect of size of specimen on reduction in area provided the shape of the cross-section remains the same (see Fig. 5).

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[1] Research Associate Professor of Engineering Materials, and Laboratory Assistant in Civil Engineering, respectively, Lehigh University, Bethlehem, Pa.

CONCERNING THE EFFECT OF NOTCHES AND LAWS OF SIMILITUDE IN MATERIAL TESTING

By A. Nadai[1] and C. W. MacGregor[1]

Synopsis

In a number of recent investigations on the various effects of stress concentration produced by notches, size effects and other discrepancies have been observed. Some of these cases are briefly analyzed and an attempt is made to include in the comparison factors such as the speed of deformation, geometrical and mechanical similitude.

A few tests made with notched bars of steel and aluminum, in which the influence of the speed of plastic deformations was considered, and photoelastic tests with notched bars are reported.

Introduction

One of the principal aims in testing materials is to derive the physical quantities determining their properties independent of all non-essential factors, such as, for example, the geometrical dimensions of the test objects. When comparing, therefore, the behavior of two pieces of the same material or of different materials having geometrically similar shapes and loaded by similarly acting forces, the question immediately arises how to define these strength properties so they will be given independently of the size of the test specimens or of other non-essential circumstances.

It will perhaps be useful to review first very briefly some known facts concerning the similitude of two pieces of a perfectly elastic material, with the view that the known circumstances in this simplest of all cases will indicate how similar relations may be discussed best in the more complicated cases encountered when plastic deformations or static rupture conditions, notch effects, etc., must be compared.

For an elastic material, the components of stress are linear functions of the components of strain. Mechanical similitude in two geometrically similar bodies of the same (elastic) material subjected to similarly applied external loads in equilibrium exists when in them the states of stress (or of strain) at corresponding points are proportional to each other. Thus if s_1, s_2, s_3 are the principal stresses in a point x, y, z in one of the two bodies and s_1', s_2', s_3' are those in the corresponding point x', y', z' in the other body, then

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[1] Consulting Engineer, and Research Engineer, respectively, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

$$\frac{s_1'}{s_1} = \frac{s_2'}{s_2} = \frac{s_3'}{s_3} = c = \text{constant} \quad \text{(1)}$$

In the absence of body forces (such as gravity, inertia forces, etc.) the external forces or the corresponding loads must be made equal to

$$Q' = c\,n^2 Q \quad \text{(2)}$$

where $n = x':x = y':y = z':z$.

A necessary, but sometimes not sufficient condition of similitude is also: the three initially perpendicular systems of curves which determine the directions of the principal stresses s_1, s_2, s_3 (or of the principal strains $\epsilon_1, \epsilon_2, \epsilon_3$) in a point (x, y, z) in the interior or at the surface of one of the two bodies and which serve to describe the state of stress (or of strain) must be geometrically similar to the corresponding system of curves of the second body; *the principal stress- (or strain-) trajectories in both bodies under stress must thus be geometrically similar.* This last method of expressing the mechanical similitude of two states of stress has perhaps the advantage that it visualizes more geometrically the relation of two states of stress. Generalization of the rule refers to such cases in which the two elastic bodies are of geometrically similar shape; however, the materials are different. If Poisson's ratio $\nu' = \nu$ for both bodies and only the elastic moduli differ ($E' \neq E$), the case is included in the former statement. If, however, $\nu' \neq \nu$ and $E' \neq E$, establishment of perfect mechanical similitude between the states of stress in two geometrically similar bodies loaded in a similar manner may or may not always be possible.[1] The presence of gravity forces adds a new condition. This is important to mention because it is a case where *body forces* are present. In the testing of materials probably one of the most important examples of this kind is the impact test. Due to the inertia forces, for example in transverse impact tests, or due to different elasticities of the supports, it may not even be possible to establish mechanical similarity in two similar cases of even perfectly elastic materials. It becomes therefore more difficult to find or to satisfy similarity relations if plastic impact has to be considered.[2]

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[1] The details cannot be explained here; it should suffice to mention two examples: (1) a photoelastic bar of bakelite having holes or notches may be a model of a similar steel bar provided the resultant of the external forces acting along each of the boundaries is zero; (2) if a flat bar is stretched by two pins fitting into two neighboring holes, a bakelite model may not strictly be a model of the similar case of a steel bar.

[2] Similar questions of practical importance have been the subject of extended investigations in connection with the design of rubber arch dam models. See A. V. Karpov and R. L. Templin, "Building and Testing an Arch Dam Model," *Civil Engineering*, January, 1932, pp. 11–16.

SIMILAR STATES OF PLASTIC FLOW

A discussion of the conditions of mechanical similitude may in a similar way be approached when in the stressed bodies permanent deformations are produced. It will be necessary to distinguish among various types of permanent or plastic strains as they may occur in metallic materials. Only metallic materials are considered.

Perfectly Plastic Material:

Under certain restrictions it may suffice to assume that: (1) The yield stresses under which a material starts to deform permanently are independent of the strains and in particular of the velocities with which the material is stretched. (2) In many cases the elastic parts of the strains can be neglected when comparing them with the permanent parts. Under these circumstances, the behavior of the material can be described by six linear stress-strain equations in which, however, one of the two material constants is now left indefinite. A condition of plasticity connecting the three principal stresses s_1, s_2, s_3 such as, for example

$$(s_1 - s_2)^2 + (s_2 - s_3)^2 + (s_3 - s_1)^2 = 2s_o^2 \ldots\ldots\ldots\ldots\ldots\ldots (3)$$

(where s_o equals yield stress in pure tension) furnishes the missing additional condition. From this last equation it follows now that in two geometrically similar bodies, when both yield, the stresses in corresponding points must either be equal or they may differ by an additive quantity. The strains may, however, differ by a constant factor in corresponding points. Unlike the case of an elastic material, the external loads required to maintain the yielding are strictly proportional to the square of the ratio of the linear dimensions (or they may differ by an additive uniform hydrostatic pressure distributed over the boundary of the body).

Strain Hardening:

The same rule applies so far as the stresses and external loads are concerned in first approximation when the yield stresses under which the plastic deformations (strains) are produced, gradually increase with these latter, that is, in those cases in which metals show strain-hardening. The elastic strains may be included in the total strains. To maintain in two geometrically similar bodies of the same material similar states of plastic strain requires that they be deformed under equal stresses (in corresponding points). This is known as the "law of proportionality" by F. Kick[1] or J. Barba.[2] The law implicitly assumes that the velocity of strain has no appreciable effect on the yield stresses. It must, however, be added that both bodies must pass through the same succession of intermediate states of stress while under loading or unloading conditions; otherwise they will not reach the same final states of strain. Two round bars of the same metal, for example, will not be in the same final states of strain if one is twisted by a couple and unloaded, the other after having passed through the same two operations in addition is twisted in the opposite sense to some extent and subsequently again unloaded.

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[1] F. Kick, "Das Gesetz der proportionalen Widerstände and seine Anwendung," **Leipzig (1885)**.

[2] J. Barba, "Resistance des materiaux, etc.," *Memoirs*, Société des Ingenieurs **Civils, Part I, p. 682 (1880)**.

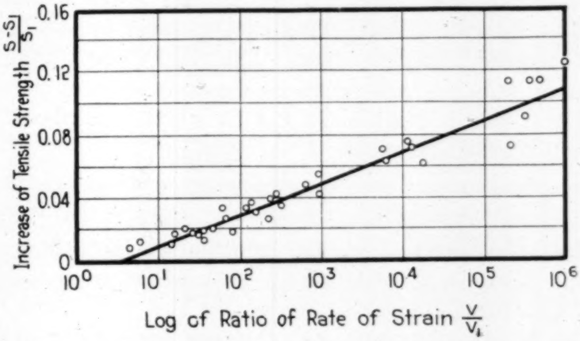

FIG. 1.—Tension Tests with Mild Steel Showing the Effect of the Speed of Stretching (Tests by H. Deutler).

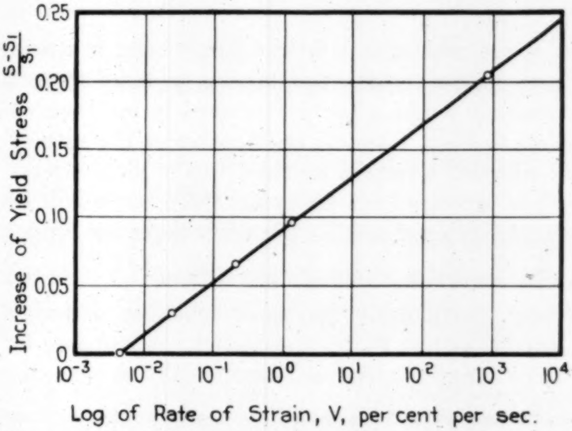

FIG. 2.—Tension Tests with Copper Showing the Effect of the Speed of Stretching (Tests by H. Brinkmann).

Effect of the Speed of Deformation:

The preceding cases referred to materials which have a definite stress-strain diagram in tension, compression or shear, such as

$$s = f(\epsilon) \qquad (4)$$

Metallic materials, particularly those having lower melting points, are known to show at normal temperatures more or less pronounced effects of the speed of deformation. The yield stress s under which a certain plastic strain ϵ was produced depends also on the rate with which the test bar is stretched or with which the plastic strain ϵ changes with time t, that is, on

$$v = \frac{d\epsilon}{dt}$$

The effect of the speed of plastic deformation, according to recent tests, seems fairly well established, at least at comparatively low testing temperatures. Figure 1 shows the test results for mild steel obtained by H. Deutler[1] and Fig. 2 those for copper obtained by H. Brinkmann[2] in tension tests. These and previous tests by P. Ludwik led to the logarithmical speed law:

$$s = s_1 + s_2 \log \frac{v}{v_1} \qquad (5)$$

where s_1, s_2, v_1 are constants. It was found valid in a wide range of velocities from a slow tension test lasting an hour up to the rates of fast impact tests in which a bar is stretched in fractions of a second.[3]

According to Figs. 1 and 2, an increase of the rate of flow by a given factor will add a certain increase to the yield stress; when the rate of flow v is increased ten times the yield stress will become 2 to 4 per cent larger for steel and 3.7 per cent larger for copper.[4]

Similitude with Regard to Speed of Deformation:

It is clear, particularly for speed-sensitive materials, that an additional condition has to be considered. Compare, for example, two bars of the same material of geometrically similar shape such as one or the other shown in Fig. 3 (*a*) or (*b*). They will have to be stretched so that at a given time t the same strains $\epsilon' = \epsilon$ are reached with the same rates of strain $v' = v$ under the same stresses $s' = s$. Thus, if their respective lengths, cross-sections and loads are l, A, P and l', A', P' then the loads P and P' at the same time t have to be made equal to P respectively $P' = \frac{A'}{A} P = n^2P$;

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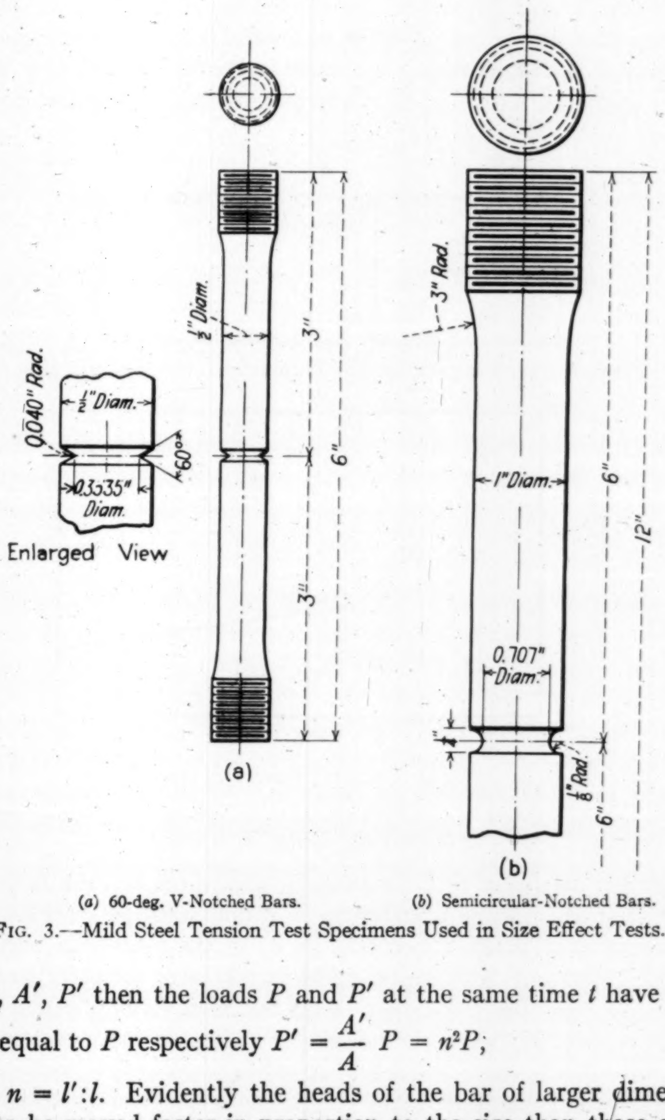
[1] H. Deutler, "Experimentelle Untersuchungen über die Abhängigkeit der Zugspannungen von der Verformungsgeschwindigkeit," *Physikalische Zeitschrift*, Vol. 33, pp. 247–259 (1932).

[2] "Zerreissversuche mit hohen Geschwindigkeiten," Dissertation, Technische Hochschule Hannover, 1933.

[3] Equation 5 evidently fails for $v = 0$ and cannot be valid for extremely slow rates of flow, which latter, however, at sufficiently low temperatures have no great practical significance.

[4] These figures show that for steel or copper under ordinary speeds of stretching such as are realized in testing machines, the effect of speed on the yield stresses is not very appreciable. However, the yield stresses observed in fast tensile impact tests may be found 10 to 20 per cent higher than in slow tests.

The conditions for mechanical similarity under impact speeds require, as mentioned, further careful considerations which cannot be included in these short remarks. It should be noted, particularly when transverse impact has to be considered, that the inertia forces due to rapid bending introduce new conditions which have to be satisfied.



(*a*) 60-deg. V-Notched Bars. (*b*) Semicircular-Notched Bars.

FIG. 3.—Mild Steel Tension Test Specimens Used in Size Effect Tests.

where $n = l':l$. Evidently the heads of the bar of larger dimension have to be moved faster in proportion to the size than those of the bar of smaller dimensions, and the load required to maintain this increased speed of the heads of the machine must be n^2 times larger at given time intervals. The ultimate strength will then be reached in both bars at the same time. As ordinarily small test specimens are tested faster than larger ones, this effect may become sensible in extreme cases of speed-sensitive materials.

NOTCHED BARS

Among the numerous tests more recently carried out in the various countries with notched bars in tension, W. Kuntze[1] particularly called attention to a pronounced size effect in such tests. He found that the ultimate strength of round bars of steel having a single and very sharp V notch (Fig. 4) in the middle of their gage lengths, in certain cases was considerably greater for quite small test specimens than for the larger ones, the bars having geometrically similar shapes, including the notch and the same notch factor:

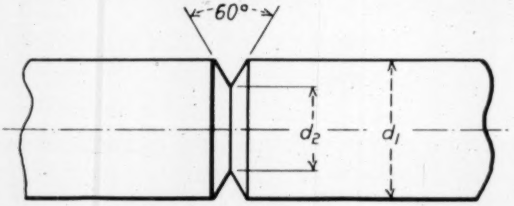


FIG. 4.—Type of Test Specimen Used by W. Kuntze. Notch factor, $K = d_2^2/d_1^2$.

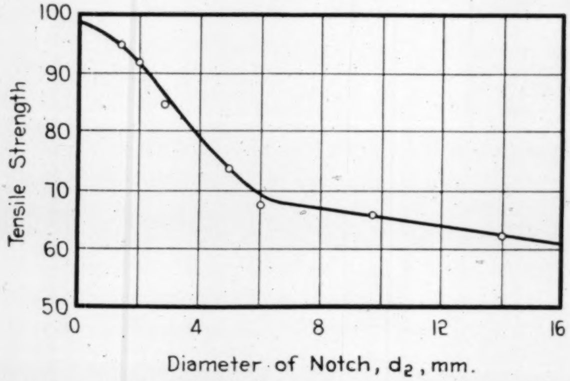


FIG. 5.—Tensile Strength of Geometrically Similar Notched Steel Bars. According to tests by W. Kuntze, $K = 0.5$.

$$k = d_2^2 : d_1^2$$

where d_1 = bar diameter, and
d_2 = smallest diameter in notch.

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[1] See W. Kuntze, "Kohäsionsfestigkeit," *Mitteilungen* der deutschen Material-prüfungsanstalten, Sonderheft 20, p. 13, Fig. 12, Julius Springer, Berlin (1932); also several articles in ***Zeitschrift für Physik***, Vol. 72, pp. 32-33 (1931).

He says, "The strength of notched test pieces does not always follow the rules of similitude, a fact which is of importance."[1]

The elastic stress concentration factors of flat or of round bars having a certain shape of notch have more recently been carefully computed and exactly determined. According to H. Neuber,[2] the maximum stress $s_{\max}$ at the bottom of the neck of a notch, the profile of which is given by the two branches of an ordinary hyperbola is approximately given by the expressions:

for flat bars.................. $s_{\max} = 1.3\, p\sqrt{a/\rho}$ (when $\rho < 10a$)

for round bars................ $s_{\max} = p\sqrt{a/\rho}$

where p is the average tension (load divided by the area in the neck of the notch), ρ the radius of curvature at the apex of the hyperbola and a the distance OA in Fig. 6. Using the second of these two formulas we find:

when $d_2 = 1.8$ mm., $a:\rho = 9$, $s_{\max} = 3p$

and when $d_2 = 6$ mm., $a:\rho = 30$, $s_{\max} = 5.5\,p$,

that is, in the case of $k = 0.5$ and the bar tested by Kuntze with the smallest neck diameter $d_2 = 1.8$ mm., half the stress concentration as in the case of a bar with $d_2 = 6$ mm. It must therefore be remarked that Kuntze's belief that his notched test bars were geometrically similar was probably not quite warranted. The sharp necks of these bars were most probably all cut with the same tool (having the same ρ for large and for small rod diameters). The larger bars were worse off than the ones with such extremely small diameters as $d_2 = 1.8$ mm.

To check these conditions, two photoelastic tests are shown here in Figs. 7 and 8, which were recently made by Mr. W. O. Richmond at the Westinghouse Research Laboratories. They were made with two bakelite flat bars, into which a notch had been cut with a depth equal to one-fourth of its width and an angle of 90 deg. The fillet radius ρ was the same for both test specimens, while the total width was respectively $\frac{3}{4}$ in. (Fig. 7) and $1\frac{3}{8}$ in. (Fig. 8). The order of the fringes indicate a stress concentration of about 8 for the smaller and about 10 for the larger width, thus showing the effect in the sense as suspected. With sharper corners the effect would have been more pronounced.

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[1] Quoted from: "Plastizität und Festigkeit bei Einkerlungen" *Zeitschrift für Physik*, Vol. 74, p. 51 (1932).

An example of Kuntze's test results is reproduced here in Fig. 5 in which the breaking stresses (computed with reference to the original cross-section in the neck of the notch $\pi d_2^2:4$) are plotted for the various bar sizes, all bars in Fig. 6 having the same notch factor $k = 0.5$. The smallest bar diameters d_2 are given in millimeters. The notches were cut with extreme care, with a very sharp tool and had "sharp" corners as indicated in Fig. 4. According to these tests his bars of $d_2 = 1.8$ mm. diameter in the notches would be nearly 50 per cent stronger than those with $d_2 = 6$ mm. or still larger notch diameters. No "size effect" was found for $k = 1$ (bar without a notch) and also for the case when k approaches zero, that is for very deep V notches. The size effect was the most pronounced when $k = 0.5$ and for only $d_2 < 6$ mm. It is believed that his results were perhaps more influenced than he probably realized or assumed by a certain lack of geometrical similarity. Since it is physically impossible to produce a sharp re-entrant corner, the notch must have had a somewhat rounded corner with a certain finite fillet. Assuming the extremely sharp fillet radius of $\rho = 0.1$ mm., which probably was scarcely reached and $a = d_2/2 = 0.9$ mm., this would make for the smallest bar the ratio of notch radius to fillet radius $a:\rho = 9$ while for the bar with a notch radius $a = 3$ mm. this ratio would be $a:\rho = 30$.

[2] H. Neuber, "Elastisch-strenge Lösungen zur Kerbwirkung bei Scheiben und Umdrehungskörper," *Zeitschrift für angewandte Mathematik und Mechanik*, No. 6, p. 422 (1933).

It is perhaps instructive to note another interesting and important result of the analysis by Neuber, namely, in the case of round tension bars with hyperbolic profiles with extremely sharp corners the stress distribution in the smallest cross-section is almost uniform with exception of a region in the closest neighborhood of the sharp, but rounded corners, where the axial stress increases to extremely high values. The stress distribution in the central portion can be described by stating that it consists of an approximately uniform axial tensile stress s_1 and a lateral uniform second tensile stress s_2 not much different from s_1 ($s_2 < s_1$). The central portions of the neck are, so to say, subjected to nearly equal tensile stresses in all directions. Yielding therefore will occur only at the corners, but due to the extreme stress concentrations these will not be able to protect the material from a very early failure or crack in tension.[1] For deep notches the stresses producing rupture will become fairly independent of the bar diameter d_1. Their effect will be to hinder the lateral contraction of

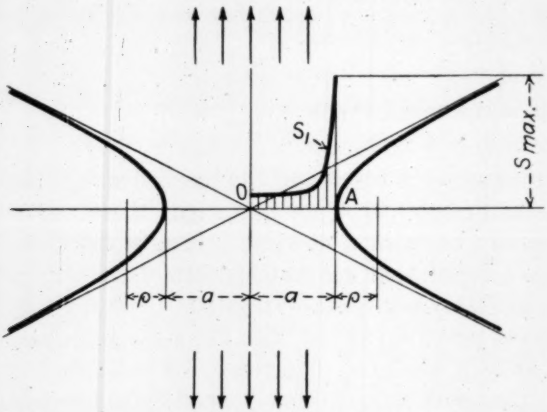

FIG. 6.—Tension Specimen with Notch of Hyperbolic Profile. According to H. Neuber, $P = \frac{\text{Load}}{\pi a^2}$.

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[1] It must be remarked that the bar profiles tested by Kuntze approach four inclined lines, which intersect at the bottom points of the notch while the asymptotes of the hyperbolic profile of Neuber intersect at the bar axis. Both cases are therefore somewhat different cases; it is believed, however, that the distributions of stress are not materially different in these two cases.

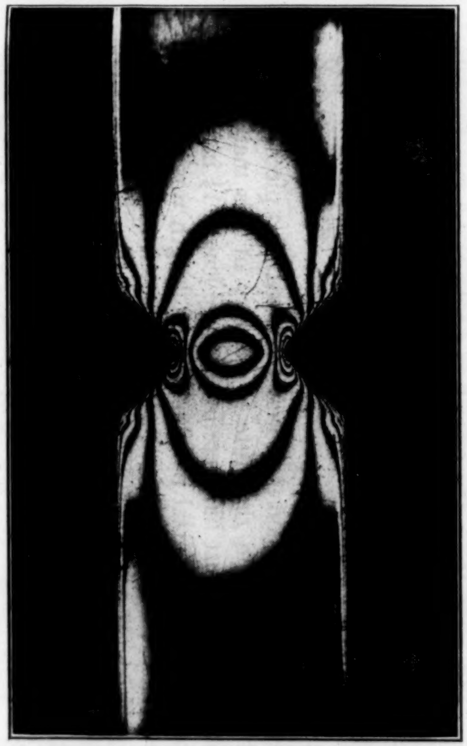

FIG. 7.—Photoelastic Test with Notched Flat Bar of Bakelite Tested in Tension.

Width of bar, $\frac{3}{4}$ in., fillet radius, $\frac{1}{16}$ in., and stress concentration factor 8.

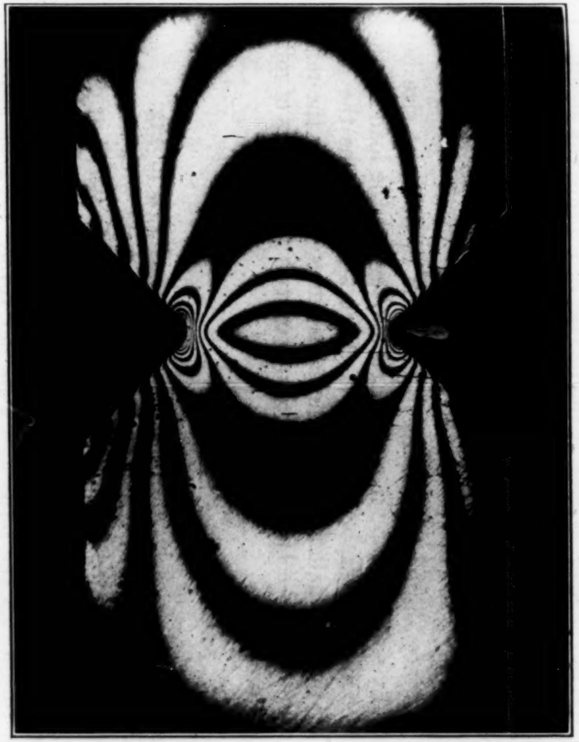

FIG. 8.—Photoelastic Test with Notched Flat Bar of Bakelite Tested in Tension.

Width of bar, $1\frac{3}{8}$ in., fillet radius, $\frac{1}{16}$ in., and stress concentration factor 10.

the material in the neck so that at the instant of rupture the smallest cross-section will not show contraction.

YIELDING TESTS WITH STRESS CONCENTRATION AT VARIOUS SPEEDS OF LOADING

To produce similar conditions in two bars of different size but geometrically similar shape it was stated above that the load on the larger piece must increase so that it is n^2 times as large as the load which acts on the smaller piece at the same times t, n being the ratio of linear dimensions. This was tested out on two sets of bars of mild steel and aluminum.

Steel:

Eight round bars having a shape such as shown in Fig. 3 (*b*) containing a semicircular notch and four round bars with a 60-deg. V notch with a rounded corner (Fig. 3 (*a*)) of various sizes but otherwise of strictly geometrically similar shapes were tested in tension till rupture. The smaller test specimens were supported near the notch in turning on the lathe and were not turned between centers. The radii of the notches were strictly proportional to the diameters. Sizes were controlled by means of a Zeiss comparator. All bars were annealed at 900 C. for one hour before machining. Table I, containing the test results, shows that although the linear dimensions of the bars varied as 8 to 1, no size effect could be detected for the ultimate strengths. In these tests, the ratio $k = d_1^2 : d_2^2 = 0.5$ was the same for which Kuntze found the maximum size effect. The smallest test specimen had a notch diameter of 0.0883 in. (=2.3 mm.) comparable with the smallest size of bars tested by Kuntze.

Aluminum:

Some additional tension tests were made with flat pieces cut from hard aluminum sheet in the state of delivery of this metal. The dimensions of the largest bar were in the gage length 1½ in. by 0.162 in. (group A). The central portion of 3 in. length was reduced to 0.125 in. thickness and contained a V type notch of 60 deg. with a rounded corner (fillet 0.0625 in.) and a width in the neck of ¾ in., the notch factor being again $k = 0.5$. Three additional bars were tested with all dimensions in the central portion reduced to ⅔, ⅓ and ⅙ of the above dimensions. Group B contained four bars of the same size as A, the depth of the V notch, however, being varied. The fillet radius ρ was left unchanged. Table II contains the results. Again no size effect was found for bars A, although the sizes varied as 6:1.

The smallest test specimen broke prematurely. As the notch depth increased (bars B) the ultimate strength increased slightly in accordance with observations of this kind by Kuntze and others, and the fact that geometrical similarity in the series B was not preserved.

TABLE I.—TENSION TESTS WITH NOTCHED ROUND BARS OF MILD STEEL.[a]
Sizes shown in Fig. 3 (*a*) and (*b*).

Test Specimen	Ratio of Sizes of Bars	Diameter at Notch, in.	Radius of Groove at Notch, in.	Average Speed of Loading, lb. per min.	Tensile Strength, lb. per sq. in.
GROUP A, SEMICIRCULAR NOTCH (FIG. 4)					
No. 1	8	0.707	0.125	3100	74 300
No. 2	8	0.707	0.125	3100	74 000
No. 3	4	0.353	0.0625	763	72 600
No. 4	4	0.353	0.0625	762	72 900
No. 5	1	0.0883	0.0156	48.4	75 900
No. 6	Broke prematurely				
No. 7	1	0.0883	0.0156	48.4	76 900
No. 8	1	0.086	0.0156	48.4	77 400
GROUP B, V-NOTCH (FIG. 3)					
No. 9	4	0.3535	0.040	763	76 800
No. 10	4	0.3535	0.040	763	75 900
No. 11	1	0.0883	0.010	48.4	76 800
No. 12	1	0.0883	0.010	48.4	75 700

[a] For all test specimens in this table the notch factor was the same: $K = 0.5$.

TABLE II.—TENSION TESTS WITH NOTCHED FLAT BARS OF HARD ROLLED ALUMINUM.

Test Specimen	Ratio of Sizes of Bars	Distance Across Bar Between Notches, in.	Average Speed of Loading, lb. per min.	Tensile Strength, lb. per sq. in.
GROUP A				
1A	6	0.750	208	19 850
2A	4	0.4998	136.8	19 450
3A	2	0.2468	22.4	19 450
4A	1	0.124	6.7	16 550
GROUP B				
	Depth of Notch			
5B	$\frac{7}{16}$	0.1237	112.5	21 200
6B	$\frac{3}{8}$	0.2471	101.5	20 400
7B	$\frac{1}{4}$	0.4998	136.8	19 450
8B	$\frac{1}{8}$	0.7513	122.3	19 300

Before closing it should at least be mentioned that in certain cases similarity relations may be disturbed by what may be called a surface effect. This phenomenon has been left out entirely from consideration in this discussion. Cast-iron pieces having a hard surface, porous materials, metallic pieces the surfaces of which have been altered (for example by rolling as it is done to improve the fatigue strengths), the presence of surface tension as demonstrated in amorphous materials in its influence on the breaking strength, the effect of a gradual disintegration of the structure due to continuous alternating stress (fatigue) and a subsequent change of the material constants in surface layers, and finally the questions related to fatigue strength need further consideration.

SUMMARY

Beginning with purely elastic materials, a few of the simplest cases of plastic deformations were reviewed with the intention of comparing the conditions under which similarity relations hold for test specimens of geometrically similar proportions. The effect of the speed of deformation for ductile metals and finally the strength of notched bars of ductile metals is discussed and some tests with notched steel and aluminum bars reported. These latter did not indicate a size effect such as has been suspected by other investigators.

EFFECT OF THICKNESS ON THE ACCURACY OF ROCKWELL HARDNESS TESTS ON THIN SHEETS

BY R. L. KENYON[1]

SYNOPSIS

The wide use of the Rockwell hardness test on sheet and strip metals makes it highly desirable to know the limitations within which the results are satisfactorily accurate. There are several variables concerned with a complete study of this problem, but this paper deals only with the effect of thickness.

This investigation has been carried out on two types of surfaces, polished and etched. Sample preparation is of utmost importance, for the material must be uniform in composition, structure, and surface condition and vary only in thickness. Low-carbon killed steel sheets free from segregation were found to be very satisfactory. They were annealed and etched to the desired thickness, then polished according to metallographic polishing technique. For the etched specimens, 0.0005 in. was then etched from each surface.

Hardness tests made on polished specimens show, in general, a constant value with decreasing thickness until a certain thickness is reached at which a bulge appears on the under surface and a lower hardness reading is obtained. This gives a series of limiting values for material of varying hardness. It is difficult to detect the first appearance of a bulge on the etched specimens, but the thickness limit in this case can be taken at the point where the Rockwell reading deviates a definite amount—say, two Rockwell numbers from the hardness of the material.

This same criterion can also be used as a practical limit on polished specimens. The obvious method of securing accurate readings on thinner specimens is to use a lighter load.

INTRODUCTION

A tentative procedure for determining the Rockwell hardness of metallic materials has been published by the Society[2], but there are several variables in the Rockwell hardness test which must yet be evaluated. Some of these variables are: thickness of specimen, smoothness of surface, lubrication of surface and ball, flatness of anvil, hardness of anvil, and roughness of ball. The present study was undertaken to throw more light on the use of this test on sheet and strip materials, especially in so far as thickness of specimen is concerned. The tests reported here were preliminary to the appointment of a subcommittee on this subject[1] and deal with ferrous materials. This subcommittee plans to extend this work to cover all commercial sheet metals.

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[1] Supervising Research Engineer, The American Rolling Mill Co., Middletown, Ohio.

[2] Tentative Methods of Rockwell Hardness Testing of Metallic Materials (E 18 - 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 967 (1933); also 1933 Book of A.S.T.M. Tentative Standards, p. 1001.

PREPARATION OF SPECIMENS

In order to study the effect of thickness on the Rockwell hardness readings on thin sheet metal, it is necessary first of all to obtain specimens of identical hardness but of different thickness. This cannot be done by cold rolling to a series of thicknesses and then annealing the specimens, even though the heat treatment succeeds in producing specimens of uniform tensile strength, since Petrenko[2] has shown that materials of the same strength may differ as much as 10 Rockwell numbers in hardness. Preparation of tapered specimens by machining may cause a variation in hardness due to cold working by the cutting tool. The proportion of the total thickness which is cold worked in this manner is greater on the thinner portions of the specimens and, therefore, affects the results. These considerations led to the development of a special technique for preparing the test specimens. The method finally adopted was to use only material of uniform composition throughout the entire thickness. Low-carbon killed steel serves this purpose admirably. Sheets of the maximum desired thickness were then heat treated to obtain uniform grain structure and hardness over their surface[3] and throughout their thickness.

Specimens were prepared by etching pieces $\frac{1}{2}$ by 4 in. in hot 1:1 hydrochloric acid until they were of the desired thickness, varying from about 0.010 in. to 0.050 in. or more by steps of approximately 0.005 in. While this etching caused no deep pits, it was necessary to polish the etched surfaces. Both top and bottom of each specimen were polished by hand with 000 emery cloth then 0, 00, and 000 Huber paper, wrapped over a flat file to polish evenly and avoid rounding the edges. X-ray examination of the surface with a glancing (10 deg.) beam[4] showed that the degree of cold working caused by the polishing was probably not more than that produced by cold rolling similar material 2 per cent. Subsequent etching and reexamination by the same method indicated that this cold-worked surface layer was not over 0.001 in. thick. It is believed that this method of preparation results in specimens of uniform hardness and smoothness of surface, and sufficient freedom from surface cold working. In order to check the uniformity throughout the thickness, a series of specimens was prepared from material 0.050 in. thick by grinding away from one side only with a special grinder used for preparing specimens for X-ray examination. Only about 0.002 or 0.003 in. were removed at a time and the hand feed was operated very slowly in order to avoid excessive heating of the specimens. They were then hand polished on both sides as described above. The hardness values duplicated those obtained on a similar series of specimens prepared by etching. This assured the uniformity of hardness throughout the thickness and also served to show that the acid etching did not affect the hardness due to hydrogen absorption.

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[1] Subcommittee on Effect of Thickness of Specimen on Rockwell Hardness Tests on Sheets and Strips (R. L. Kenyon, chairman) of Section on Thin Sheet Testing of Committee E-1. The chairman invites correspondence on this subject.

[2] S. N. Petrenko, "Relationship Between Rockwell and Brinell Numbers," National Bureau of Standards *Research Paper No. 185*, Vol. 5, July, 1930.

[3] In order to test the uniformity over the surface, about 20 pieces $\frac{1}{2}$ by 4 in. were cut in succession and numbered consecutively. Five Rockwell impressions were made on the alternate pieces. When the averages of these all fell within the limits $\overline{X} \pm 3\,\sigma_{\bar{x}}$, the material was considered to be uniform. For details, see W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., Inc., New York City (1931). The test specimens were prepared from the alternate pieces.

[4] L. Thomassen and D. M. McCutcheon, "Use of X-ray on Depth of Cold Work by Machining," paper presented at New York meeting, Am. Soc. Mechanical Engrs., December, 1933.

The specimens tested with an etched surface were prepared exactly as those described above with the additional step of a final etching treatment in the hot hydrochloric acid which removed 0.0005 in. from each surface. This gave a constant depth of etching and the uniformity of the surface from piece to piece was much better than after the first etching.

This technique has been described in detail because it is believed that these or similar precautions must be taken to eliminate all other variables except thickness.

METHOD OF TESTING

Several series of specimens were prepared according to the above technique and on most of them impressions were made with several different loads. Five similar impressions were made on each test specimen and the average taken as the hardness reading. It was found that these must be spaced far enough apart so that no part of a bulge under an adjacent impression rests on the "spot" anvil, and so that the cold-worked area around the top of the adjacent impression does not affect the results. The hand-polished specimens had slightly rounded edges and it was necessary to make the impressions near the midwidth. The 15, 30, and 45-kg. loads were applied with a new model 1-S superficial Rockwell tester loaned by the Wilson Mechanical Instrument Co., for making these tests, and the 60, 100, and 150-kg. loads with a new model 3-M standard Rockwell tester. The penetrator in all cases was a $\frac{1}{16}$-in. ball of the kind supplied with the testing machines. A new steel "spot" anvil was used and when damaged through testing some of the thinner specimens, it was resurfaced by metallographic polishing, using a protecting ring to avoid rounding-off the "spot." The A.S.T.M. Tentative Methods for Rockwell Hardness Testing (E 18 - 33 T) were followed for the standard tester and the manufacturers' method for the superficial tester.

TABLE I.—AVERAGE ROCKWELL READINGS ON SERIES C SPECIMENS.

100-kg. load, $\frac{1}{16}$-in. ball, 5 observations on each thickness.

Specimen	Thickness, in.	Average, $\overline{X}$	Standard Deviation, σ	Description of Impression
C-1	0.047	46.18	0.3374	Prominent cone. Polished bottom.
C-2	0.0425	45.38	0.4308	Prominent cone. Slight bulge. Boundaries irregular.
C-3	0.039	44.36	0.2870	Cone decreasing. Bulge larger, round and well defined.
C-4	0.035	44.32	0.2712	Cone decreasing. Bulge higher, diameter smaller.
C-5	0.0305	43.80	0.2097	Cone decreasing. Bulge higher, diameter smaller.
C-6	0.0265	43.10	0.4146	Cone decreasing. Bulge higher, diameter smaller.
C-7	0.0215	43.04	0.3929	Depression. Bulge higher, diameter smaller.
C-8	0.016	41.40	0.5656	Depression. Bulge higher, diameter smaller.
C-9	0.012	44.16	1.5965	Depression. Bulge higher, diameter smaller.
C-10	0.0085	42.26	1.1898	Depression. Bulge higher, diameter smaller.

TABLE II.—LIMITING THICKNESS OF SHEETS FOR ACCURATE ROCKWELL READINGS.

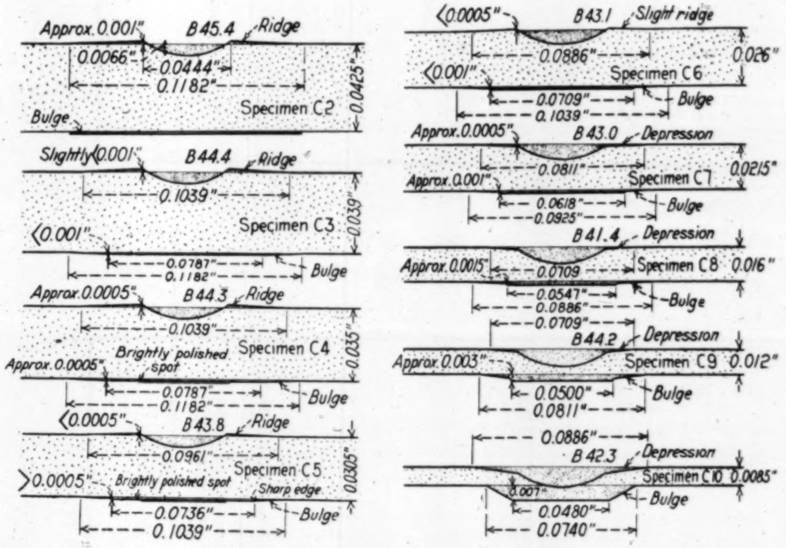
Material Designation	Rockwell Tester[a] Used	Load, kg.	Rockwell Scale and Hardness Number	Nominal Depth of Impression, in. (d)	Maximum Thickness on Which Bulge Occurred, in. (T)	Ratio, T/d	Thickness Greater Than and Less Than That at Which Rockwell Value Deviates Two Numbers from That of Thickest Specimen, in.		
							Next Thicker	Next Thinner	Mean
POLISHED SPECIMENS									
J	S	15	15T-76	0.00096	0.021	21.90	0.021	0.0168	0.019
J	R	100	B-45	0.0068	0.0415	6.1	0.039	0.031	0.035
E	R	100	B-89	0.0033	0.029	8.79	0.029	0.025	0.027
C	R	60	F-83.8	0.0037	0.035	9.46	0.0265	0.0215	0.024
C	R	100	B-46.2	0.0067	0.0425	6.35	0.035	0.0305	0.033
O	R	60	F-82.4	0.0038	0.0286	7.53	0.0286	0.027	0.0278
O	R	100	B-41.8	0.0071	0.0425	5.98	0.039	0.0286	0.034
ETCHED SPECIMENS									
N	S	15	15T-72.5	0.0011	......	.....	0.0395	0.033	0.0362
N	S	30	30T-39.5	0.0024	......	.....	0.046	0.0395	0.0432
P	R	100	B-91	0.0031	......	.....	0.0495	0.0435	0.0465
P	R	150	G-67.5	0.0050	......	.....	0.060	0.0495	0.055
M	R	100	B-56	0.0059	......	.....	0.061	0.051	0.056
M	R	150	G- 9.7	0.0096	......	.....	0.081	0.069	0.075

[a] S = Superficial Rockwell tester. R = Regular Rockwell tester.

After the specimens were tested, the appearance of the surface around and under the impressions was studied with a pocket magnifier and a binocular microscope fitted with filar eyepiece. The specimens were rubbed lightly with 0000 Huber paper as an aid to determining the nature of the flow around and under the impressions. It was difficult to do this on the etched specimens on account of the roughness of the surface and these specimens, although carefully examined, have not been graded according to this criterion.

RESULTS

It is obviously impossible to present all of the numerical results within the limited space devoted to this paper, but Table I shows the form in which the data were compiled. These results were obtained on polished specimens of low-carbon killed steel. Figure 1 is based on a minute examination of the impressions and measurement with a filar eyepiece in the binocular microscope. Specimen C-1, not included in the illustration, showed a ridge on the top side around the ball indentation. There was no bulge on the underside. Attention is called to the bulge on the underside of the impression on specimen C-2.



Approx. 0.001"
B 45.4
Ridge
0.0066"
0.0444"
0.1182"
Specimen C2
0.0425"
Bulge
Slightly <0.001"
B 44.4
Ridge
0.1039"
Specimen C3
0.039"
<0.001"
0.0787"
0.1182"
Bulge
Approx. 0.0005"
B 44.3
Ridge
0.1039"
Brightly polished spot
Specimen C4
Approx. 0.0005"
0.035"
0.0787"
0.1182"
Bulge
<0.0005"
B 43.8
Ridge
0.0961"
Specimen C5
>0.0005"
Brightly polished spot
Sharp edge
0.0305"
0.0736"
0.1039"
Bulge
<0.0005"
B 43.1
Slight ridge
0.0886"
0.026"
<0.001"
Specimen C6
0.0709"
0.1039"
Bulge
Approx. 0.0005"
B 43.0
Depression
0.0811"
Specimen C7
0.0215"
Approx. 0.001"
0.0618"
0.0925"
Bulge
B 41.4
Depression
Approx. 0.0015"
0.0709
Specimen C8
0.016"
0.0547"
0.0886"
Bulge
0.0709"
B 44.2
Depression
Approx. 0.003"
Specimen C9
0.012"
0.0500"
0.0811"
Bulge
0.0886"
B 42.3
Depression
Specimen C10
0.0085"
0.007"
0.0480"
0.0740"
Bulge

FIG. 1.—Deformation Around Rockwell Impressions on Specimens of Different Thickness.

It is rather difficult to describe the appearance of this bulge and efforts to photograph it have not been successful due to the fact that it is best seen by "gun-sighting" the specimen toward a light source. On the thickest specimens showing the very first appearance of a bulge on the underside, it is generally of somewhat irregular outline. Thinner pieces show well-rounded bulges. When the bulge is barely visible and irregular, the hardness reading is generally reliable. This is

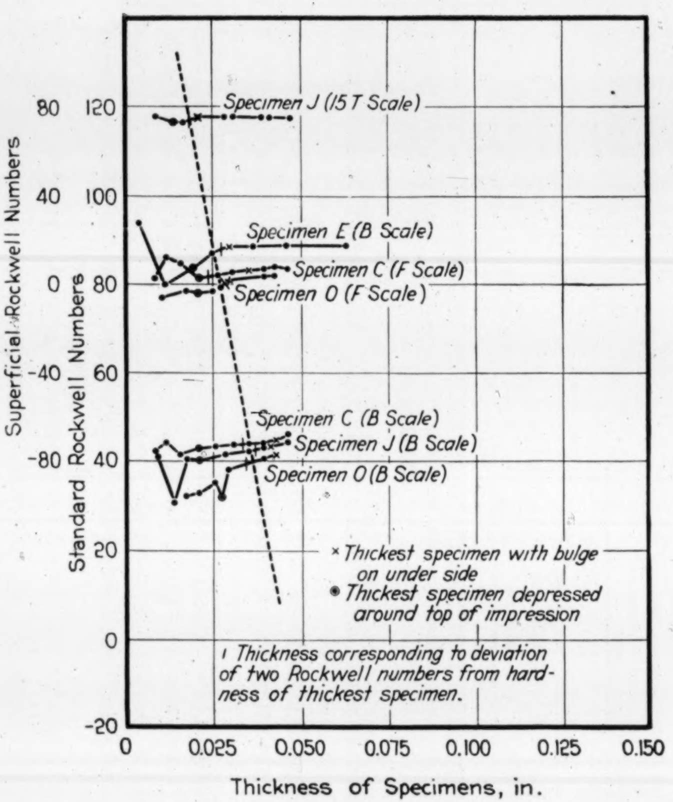


FIG. 2.—Variation in Rockwell Readings with Thickness on Specimens with Polished Surfaces.

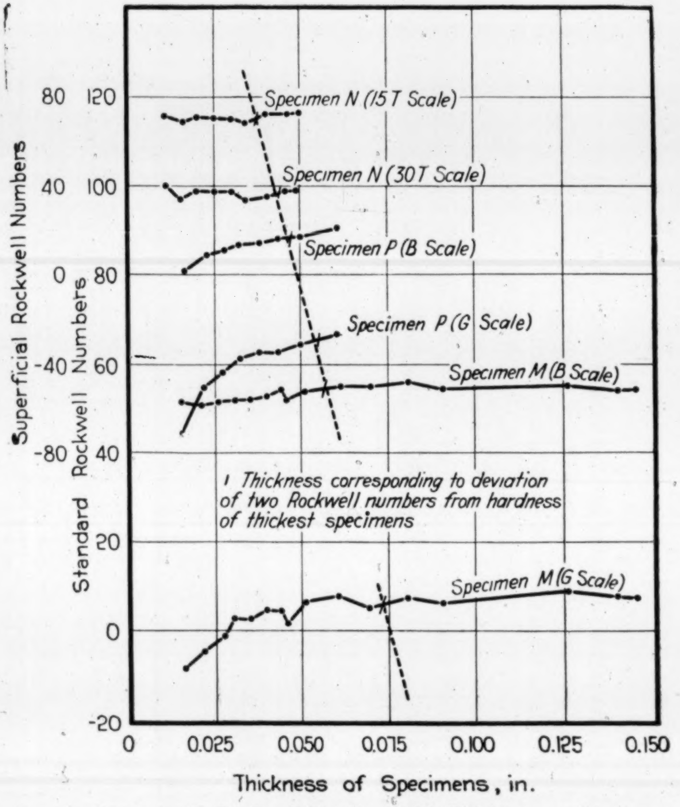


FIG. 3.—Variation in Rockwell Readings with Thickness on Specimens with Etched Surfaces.

thought to be due to the fact that the lateral flow under the ball is just beginning and is insufficient to cause any appreciable increase in depth of indentation or downward movement of the penetrator. The prominence of the bulge increases as the thickness diminishes on successive specimens and the ridge on the top around the spherical depression decreases in height until on specimen C-6 it is barely discernible. On C-7 and thinner specimens, there is a depressed area around the spherical indentation.

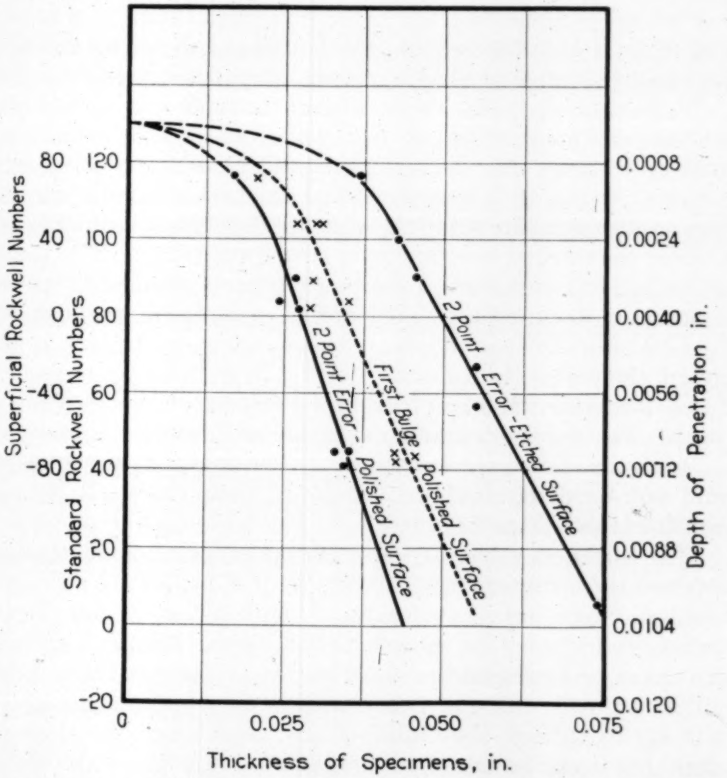


FIG. 4.—Minimum Thickness and Rockwell Hardness Impressions on Material of Different Hardness as Judged by a Difference of Two Rockwell Numbers from the Hardness Reading on the Thickest Specimen Tested and As Judged by the Appearance of a Bulge Under the Impression on Specimens with Polished Surfaces.

The hardness decreases from the thickness at which the bulge appears on the underside until the ridge on top changes over to a depression. Near the thickness which corresponds to that at which the depression on the topside appears, there seems to be a rather narrow range in which anvil effect (higher readings) occurs. After this, further decrease in thickness causes "punching through" of the penetrator and a corresponding low reading. This is due to insufficient lateral stiffness and the metal "buckles." On some series the anvil effect is not observed and this is thought to be due to the narrowness of the range in which the anvil effect is detected.

Values corresponding to those given in Table I were obtained for each series of impressions on the various specimens. The average hardness readings for the polished specimens are plotted against thickness in Fig. 2. In general the thickest specimen showing a bulge on the underside is the thinnest one that gives accurate hardness values or the thickest one that gives erroneous results. This furnishes one method of determining a limiting thickness of polished specimens for accurate Rockwell tests on material of different hardness, or for impressions of different depth. The slanting dotted line is drawn through the points on the various curves, corresponding to deviations of two Rockwell numbers[1] from the hardness of the thickest piece tested. This furnishes another criterion of limiting thickness. The results on the etched specimens are plotted in Fig. 3, but since bulges could not be determined accurately on them, only the two-point error line is shown on this chart.

The thickness values at which the bulge occurs on polished specimens and those corresponding to two-point errors for the polished and etched specimen are plotted against depth of impression (Rockwell number) in Fig. 4. The superficial and regular Rockwell scales have been taken to correspond to equal depths of penetration so that the results obtained with the two machines may be compared.[2] It is quite apparent that the shallower the impression, the thinner the material that can be tested without causing a bulge on the underside or an error greater than a given amount, say two Rockwell numbers. These shallower impressions may be the result of either greater hardness of the material tested or of lighter loads on the penetrator. The scatter of the points is due partly to the limited number of materials and loads used. The variation in thickness in the test specimens was not continuous but by increments and the *exact* location of the points where the bulge and the two-point error first occurs was therefore not possible. It is believed that the curves represent the average results. While no impressions have been made shallower than about 0.0009 in , it is quite evident that the curves must pass through the point corresponding to zero indentation at zero thickness and they are so shown as dashed lines.

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[1] This deviation of two Rockwell numbers was used because experience has shown it is hardly practical to hold several machines in plant inspection laboratories within any closer limits than ± two Rockwell numbers from the average hardness of standard blocks furnished by the manufacturer, in the range B-40 to B-70. These limits may be less than this for harder materials.

[2] The minor load on the superficial tester is 3 kg., while on the regular tester it is 10 kg. This makes impossible direct comparison of hardness values obtained on the two machines, but the scales plotted in Fig. 4 approximately represent the depth of the impressions made with the two machines

It will be noted that the two-point error curves in Fig. 4 indicate a greater limiting thickness for specimens with etched than with polished surfaces. This is thought to be due to the greater flow of metal into minute surface cavities and pits on the etched surfaces. The appearance of the bulge on the polished specimens furnishes a more exact limit of thickness for accurate results and requires a thicker specimen than the two-point error limit. The latter is probably of greater practical utility.

DISCUSSION

It is realized that the data presented are too limited to justify any sweeping generalizations. The principal object of this paper is to call attention to the need for definite limits for thickness of specimens for Rockwell tests and to indicate a method of approaching the problem. Sample preparation is believed to be so important that considerable space has been devoted to this in the hope that others who may carry out these tests on other metals will observe the precautions that have been described.

The data obtained indicate that it should be possible to establish definite minimum values for thickness for materials having definite surface characteristics, but additional work is needed, particularly on other materials and surfaces than those included in these tests. The appearance of a bulge under the impression is believed to be a sensitive indication of the accuracy of the Rockwell readings, but this can only be applied to fairly smooth surfaces because it is obscured by surface pits, etch pattern, etc. This limit, which may be of academic and theoretical interest, is at a somewhat greater thickness than that which permits a maximum deviation of two Rockwell numbers from the hardness of the material.

The curves in Fig. 4 giving the two-point error limit for the polished and etched surfaces show the *important influence of surface condition on the hardness reading*. These curves will doubtless have to be modified to apply to other materials and surfaces, but are given here to show how limits for thickness might be stated.

In conclusion, a word of warning must be given against making too broad an application of the results of these tests to cases occurring in practice. A method has been outlined which the author hopes many will apply to materials other than those with which he was concerned. It is only through such additional experimentation that generalizations can be made regarding the effect of thickness on the accuracy of Rockwell impressions.

SUMMARY

In the preparation of specimens for this test, a low-carbon killed steel was used of uniform composition and structure throughout its thickness. Variation in thickness was obtained by etching in acid and the surface was then polished.

The appearance of a bulge under an impression on a polished specimen is evidence that such a Rockwell reading is questionable. The thickest specimen showing this is either the thinnest one that gives accurate hardness values or the thickest one that gives erroneous results due to thickness effect. The appearance of a bulge cannot be used as a criterion on etched surfaces and its application to polished specimens is principally of theoretical value. It is possible to determine a practical limiting thickness for various surfaces which will insure a maximum deviation of say two Rockwell numbers from the hardness of the material.

On iron and steel the ridge around the spherical impression changes to a depression at a certain thickness of the specimen.

There are apparently three influences that affect the Rockwell hardness reading on thin sheets; side flow causing lower readings, anvil effect causing higher readings, and crushing effect or "punching through" causing lower readings. The intensity of these effects varies with thickness, and the reading obtained is the net result of all three superimposed on the hardness of the material.

Decreasing the load (changing the Rockwell scale) makes it possible to obtain reliable readings on thinner pieces.

Acknowledgments:

The author expresses his appreciation to the Wilson Mechanical Instrument Co. for the loan of a superficial Rockwell tester and for supplying special test blocks used in this work. Messrs. R. O. Griffis and R. F. Passano, of the Research Laboratories, made numerous helpful suggestions in the interpretation of the results and Mr. Harry Tobin, of the same laboratories, rendered valuable assistance in the preparation and testing of specimens and in analyzing the data.

DISCUSSION

Mr. C. H. Marshall[1] (*presented in written form*).—Mr. Kenyon's conclusion that accurate hardness readings are obtained when there is not more than a barely discernible "bulge" on the underside of the specimens is a confirmation of a general fact which long has been known in connection with Brinell hardness testing. His findings should be a caution to those who have been inclined to consider hardness tests made on thin sheets to represent the actual hardness of the material.

This, however, does not mean that hardness tests of great value cannot be made on even very thin sheets. It is evident that for similar material of the same thickness the errors introduced by what might be spoken of as the "thinness effect" should, in general, be of the same order. Therefore, for comparing the hardness of similar thin materials of the same thickness, penetration hardness tests such as Rockwell or Vickers, should be useful. In this connection, I suggest that in reporting results of tests made on any material sufficiently thin that even a slight "bulge" is likely to be produced, the thickness of the material as well as the pressure applied and the kind of penetrator be made an essential part of the hardness result as a warning that this figure does *not* represent actual hardness of the material but is merely a figure useful for comparison with other tests made under the same conditions with respect to penetrator, pressure, thickness, etc.

At first thought, one might expect that the error when very thin materials are tested would be in the direction of increasing hardness caused by the effect of the hard anvil. But as pointed out in this paper, while under certain conditions there is some effect of this kind, it is almost completely overshadowed by other conditions which cause the readings to be low rather than high.

On tests of phosphor bronze and some other non-ferrous materials in various thicknesses which I made some time ago, this decrease in hardness was noticed from about the time that the "bulge" appeared on the underside of the specimen, then an apparently greater decrease in hardness for thinner samples with a somewhat lessened decrease of hardness at some point as the thickness was further decreased (anvil effect), then the very much lowered readings as the material buckled and the penetrator "punched through."

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[1] Materials Engineer, Lucius Pitkin, Inc., New York City.

In the series of tests which I made, anvils of various materials (including diamond) were used and it was found that for these extremely thin materials, the surface condition of the anvil and also the material of which it was made influenced the hardness readings. It was therefore concluded that the coefficient of friction between the anvil and the material being tested affected the hardness readings as also would the presence or absence of any lubricant on the underside of the material or its surface condition.

The curves of Fig. 4 of the paper are very interesting and give what might be considered as some absolute data on limiting thickness of material on which accurate hardness readings may be obtained. I believe that when sufficient similar data are available for various materials, an equation can be developed for defining the maximum allowable penetration for any material of any thickness and hardness.

MESSRS. C. H. DAVIS[1] and R. S. BAKER[1] (*presented in written form*).—Mr. Kenyon's investigation of the Rockwell hardness of the thin sheets has been confined to that of low-carbon killed steel. To widen the field of study and obtain corroborative data from nonferrous alloy sheets, specimens were prepared of nickel silver and brass of the following approximate compositions:

	COPPER, PER CENT	ZINC, PER CENT	NICKEL, PER CENT
Nickel silver..........................	56.00	26.00	18.00
Brass..............................	72.00	28.00	....

The material selected was soft, 2 and 8 B.&S. numbers hard, 0.040 or 0.065 in. thick and 6 in. wide.

In the case of the nickel silver, three samples 1 by 4 in. were taken from the central portion of the bar with the 4-in. dimension in the direction of rolling. With the brass, two samples were taken in a similar manner.

After the original hardness was determined, the samples were reduced in thickness by etching in 10-per-cent nitric acid solution. After etching, the surface of the specimens was polished lightly with No. 0 emery cloth until the surface was smooth. Then the specimens were placed for ten minutes in a sodium dichromate dip,[2] and dried. This yielded a smooth surface with most of the polishing scratches etched off.

By testing near the center of the strip, the variation over the test width was not more than 0.001 in.

This procedure was repeated a number of times on each sample until the samples had been reduced in thickness to 0.010 in. or less.

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[1] Technical Dept., The American Brass Co., Waterbury, Conn.

[2] Sodium dichromate dip.—2000 ml. of a cold saturated $Na_2Cr_2O_7$ solution, 40 ml. of a cold saturated NaCl solution, and 200 ml. concentrated H_2SO_4.

Three impressions were made on each gage, $\frac{1}{4}$ in. apart and in a line across the strip. On each succeeding thinner gage three similar impressions were made in a line $\frac{1}{4}$ in. from the last, so that there was no danger of any one series of impressions being made over another.

In the case of the soft brass and nickel silver, the Rockwell "F" scale appears accurate to about 0.040 in. thickness, while in the case of the "B" scale the accuracy on the brass is affected at about 0.060 in., and on the nickel silver at about 0.040 in. With the "G" scale soft nickel silver may accurately be tested to about 0.050 in.

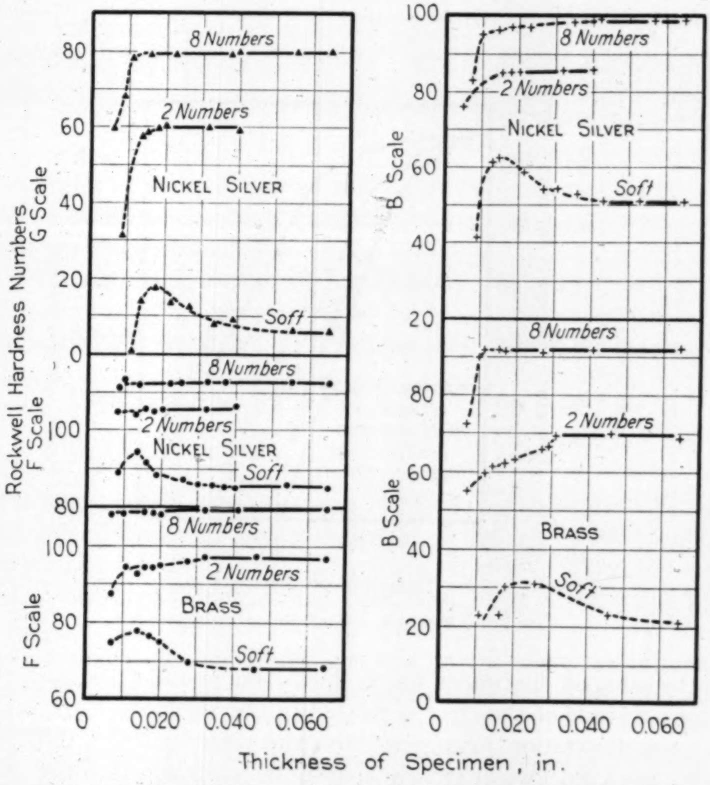

FIG. 1.—Rockwell Hardness Numbers of Nickel Silver and Brass.

Similar observations for hard brass and nickel silver may be drawn from the accompanying Fig. 1. The harder specimens may be accurately tested to gages in the range from 0.020 to 0.010 in. thick.

MR. R. L. KENYON[1] (*author's closure by letter*).—The discussion by Mr. Marshall raises a point in regard to the value of indentation hardness tests on specimens of the same thickness when a "bulge" is produced under the impression. Mr. Marshall considers the hardness readings comparable if the specimens are of equal thickness. The author does not agree with this. In Fig. 2 of the paper a line has been drawn to show the thickness corresponding to a deviation of two Rockwell numbers from the hardness of the thickest specimen. Other similar lines could be drawn to the left of this one corresponding to deviations of three, four, or five Rockwell numbers. These lines would intersect an ordinate corresponding to a given thickness at lower and lower hardness values. It is thus seen that although the thickness may be the same on specimens which show a bulge under the impressions, the magnitude of the error is related to the hardness of the material.

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[1] Supervising Research Engineer, The American Rolling Mill Co., Middletown, Ohio.

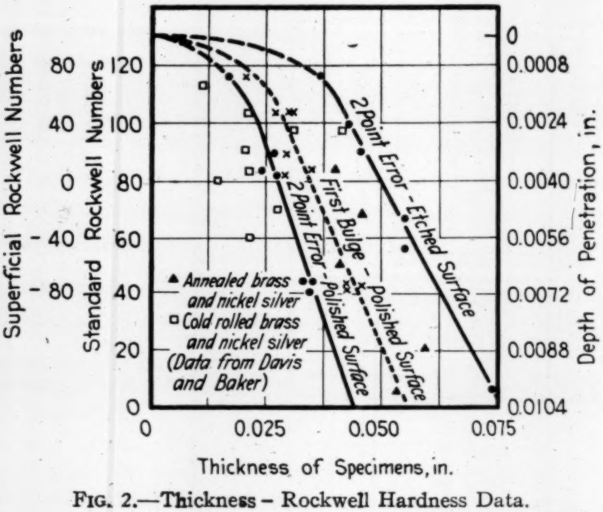

FIG. 2.—Thickness – Rockwell Hardness Data.

For illustration, assume we have two pieces 0.025 in. thick, one of which has a Rockwell hardness of 100 "B" scale and the other 30 "B" scale. The surface condition may be identical on the two pieces, but the results on the softer one will be considerably in error while the harder one would give essentially correct hardness readings.

It should also be remembered that, so far, data have been obtained only for low-carbon ferrous metals (and brass and nickel silver as contributed in the discussion by Messrs. Davis and Baker). The location of lines for errors of 2, 3, 4, and 5 Rockwell numbers may be different on other metals. This is another argument against the belief that hardness readings are comparable on specimens of the same thickness regardless of the material, its hardness, or the presence of a bulge under the impression. There is only one remedy when the impression penetrates through the specimen and that is to reduce the load so that the depth of the impression bears the proper relationship to the thickness. This relationship is given for low-carbon steel in Fig. 4 of the paper.

The data presented by Messrs. Davis and Baker offer a valuable contribution to that given in the paper. There are a number of interesting observations that can be made from their Fig. 1. In the first place there seems to be a marked difference in the behavior of their annealed and cold-rolled samples. The soft specimens gave higher hardness readings with decreasing thickness while the opposite was true of the cold-rolled pieces. The numerical data for Fig. 1 are not given and there is no statement of the criterion used to determine the limiting thickness for accurate Rockwell readings, but it is presumed that this has been taken where the readings have deviated a set amount from that of the thickest specimen.

When the thickness values corresponding to the left end of each *solid* line in their Fig. 1 are plotted as in Fig. 4 of the paper, the accompanying Fig. 2 is obtained. This shows that there is good agreement for brass, nickel silver and low-carbon steel *if the material is in the annealed condition*. There is a wide scatter of the points for the cold-rolled brass and nickel silver and this suggests the possibility that the etching away of the cold-rolled surfaces has affected the results. This does not necessarily mean that the limiting thickness given in Fig. 4 would not apply to cold-rolled material; it merely means that when the surface of cold-rolled material is removed, as by etching, the resulting strip does not behave in the same manner as an annealed piece. The data given by Messrs. Davis and Baker indicate that the brass and nickel silver which they tested conform to the same limitations for accurate Rockwell tests as the low-carbon steel tested by the author, provided the material is in the annealed condition.

A subcommittee of the Thin Sheet Testing Section of the Society's Committee E-1 on Methods of Testing hopes to expand this work to cover all of the commercial sheet materials so that this information will be available as a guide as to the minimum thickness upon which reliable Rockwell readings can be made. In closing, the author expresses his appreciation of the additional data that have been presented and makes a plea that those interested in other metals conduct similar series of tests and report their results to the committee.

STUDY OF CEMENT COMPOSITION IN RELATION TO STRENGTH, LENGTH CHANGES, RESISTANCE TO SULFATE WATERS AND TO FREEZING AND THAWING, OF MORTARS AND CONCRETE

By H. F. Gonnerman[1]

SYNOPSIS

This paper presents data obtained over a period of three years in an extensive cooperative investigation between the Research Laboratory of the Portland Cement Association, Chicago, and the Portland Cement Association Fellowship at the National Bureau of Standards, Washington.

In this investigation 121 laboratory-prepared portland cements, covering 72 different compositions and ground to essentially the same fineness, were studied in relation to strength, length changes, resistance to sulfate waters and to freezing and thawing, in mortars and concrete. In this paper the results of tests on 74 of the cements are presented which include several compositions covering the effect of variations in MgO content.

The results reveal that mortar and concrete properties are markedly influenced by the compound composition of the cement. The effects of each of the four major cement compounds on the various properties investigated are discussed.

In order to develop a better understanding of the constitution and properties of cement, the Portland Cement Association through its Fellowship at the National Bureau of Standards has, since 1924, been conducting a broad program of fundamental research. As one phase of this work there was inaugurated in 1930 a cooperative series between the Fellowship and the Research Laboratory at Chicago, designed to study the influence which the various compounds known to exist in cement have on its properties. This series was planned as an exploratory investigation with a view to following up with more extensive studies any important trends which the preliminary tests might disclose. While the tests are not completed, they have progressed to a point where significant results have been obtained. The keen interest in the nature of the compounds existing in cement, in the influence which these compounds have on the properties of the cement, and in the development of specifications for special portland cements makes the presentation of the data particularly timely.

This paper presents only the results of tests made at the Research Laboratory in Chicago on the cements prepared in the course of the investigation.[1] Tests made by the Fellowship at Washington on the same cements have been reported elsewhere by Bogue and Lerch(1,2).[2]

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[1] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

Acknowledgments.—The cements used in the investigation were prepared by the Portland Cement Association Fellowship at the Bureau of Standards. This Fellowship is under the immediate supervision of R. H. Bogue, and under the general direction of P. H. Bates, Chief, Clay and Silicate Products Division of the Bureau. The cements were prepared, burned and ground, as described below, by W. C. Taylor of the Fellowship staff. Mr. Taylor also made analyses for uncombined lime and SO_3. Acknowledgment is made of the generous assistance of the Bureau of Standards, and of the valuable cooperation of Mr. Bogue and his associates in the planning of the investigation and in the manufacture of the cements. Chemical analyses of raw materials, raw mixtures and clinkers were made at the Research Laboratory, Chicago, by C. L. Ford. The author is indebted to A. G. Timms, G. W. Ward, W. R. Johnson, T. G. Taylor, L. A. Dahl, W. A. Ebert, and to other members of the staff who participated in the conduct of the tests, for their assistance in the assembling and preparation of the data for this report.

MANUFACTURE AND COMPOSITION OF CEMENTS

One hundred and twenty-one cements covering 72 different compositions were prepared from commercial raw materials for use in the tests. The range of compositions was purposely expanded beyond that of normal portland cements. The comprehensive program and the limitations of equipment and personnel necessitated extending the manufacture of the cements and the fabrication of the test specimens over the period from April, 1930, to November, 1933.

The raw materials consisted essentially of limestone and clay. In a number of cases, however, flint, bauxite, iron ore or dolomite were introduced in order to bring the mixtures to the desired compositions.

The raw materials were ground to a fineness of 95 per cent through the No. 200 sieve, formed into $\frac{3}{16}$ to $\frac{7}{16}$-in. balls which were burned in a gas-fired rotary kiln, 5 in. in internal diameter and 8 ft. long, having a capacity of 10 to 15 lb. of clinker per hour. The temperature of burning for complete combination of the various raw mixes was predetermined by means of auxiliary tests in a small electrically heated furnace. The proper burning temperature and rate of burning to give substantially complete combination was then maintained during the burning of the various clinkers in the rotary kiln. Where a high content of uncombined lime was indicated, the clinkers were double-burned. After burning, the clinkers were cooled in an open can, passed through rollers and then ground in an 18 by 30-in. ball mill with the proper amount of gypsum to give a constant SO_3 content in the cement. A portion of the ground cement was retained for test at the Fellowship and the remainder was shipped to Chicago in sealed metal cans for the various tests described in this paper.

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[1] Attention is called to the fact that although the cements used in this study were prepared at the National Bureau of Standards, the making and testing of the specimens and the preparation of this paper were carried out by the staff of the Research Laboratory of the Portland Cement Assn. at Chicago. The discussion of the data and the conclusions presented herein should not be interpreted as reflecting the viewpoints of the Bureau.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 272.

The oxide compositions of the different cement clinkers and their compound compositions computed according to the methods outlined by Bogue(3) are given in Tables I and XI. In these tables and in the text the cement compounds are abbreviated as follows:

COMPOUND	ABBREVIATION
Tricalcium silicate	C_3S
Beta dicalcium silicate	C_2S
Tricalcium aluminate	C_3A
Tetracalcium alumino ferrite	C_4AF
Dicalcium ferrite	C_2F

The cements were prepared systematically in groups and subgroups in accordance with a predetermined outline covering the variables under study as follows:

Group 1: C_3A decreases; C_3S constant; R_2O_3 ($Fe_2O_3 + Al_2O_3$) constant.
Group 2: C_3A decreases; C_3S increases; R_2O_3 constant.
Group 3: C_3A decreases; C_3S constant; R_2O_3 increases in each of six subgroups and decreases from subgroup to subgroup.
Group 4: C_3A decreases; C_2S and C_3S decrease; C_3S/C_2S constant; R_2O_3 varies.
Group 5: C_3A constant; C_3S and C_2S decrease; C_3S/C_2S constant; R_2O_3 increases.
Group 6: (*a*) MgO substituted for CaO; C_3S decreases.
(*b*) MgO added; C_3S constant; C_2S decreases.
Group 7: C_3S increases in each of six subgroups; C_3A decreases from subgroup to subgroup; R_2O_3 constant.
Group 8: C_3S constant in each of three subgroups; C_3A decreases from subgroup to subgroup; SO_3 contents, 1.0, 1.8, and 2.6 per cent.
Group 9: C_3S increases and C_3A constant in each of three subgroups; 0, 1, 3, 5, 10, and 15 per cent additions of precipitated silica by weight of cement.

Because of lack of space, results for the cements in Groups 8 and 9, above, are not included here but will be presented in later reports.

For convenience of study on the basis of compound composition, the cements in Table I, and in succeeding tables except IX and XI, have been arranged in six groups according to their computed C_3S contents as follows:

(1) Computed C_3S above 60 per cent
(2) Computed C_3S 51 to 60 per cent
(3) Computed C_3S 41 to 50 per cent
(4) Computed C_3S 31 to 40 per cent
(5) Computed C_3S 21 to 30 per cent
(6) Computed C_3S below 21 per cent

In each of the foregoing groups the cements are arranged in descending order of C_3A content, which in some of the groups varied from 0 to as much as 20 per cent. The latter value is considerably higher than the average C_3A content of present-day commercial portland cements.

All the cements in Table I had the same SO_3 content, 1.8 per cent by weight, and in general contained the same amount of MgO, approximately 3 per cent by weight. Table XI gives the composition of other groups of cements in which MgO content was the principal variable studied. The values for the chemical analyses are the average of 2 determinations. The analyses in most cases were made on the clinkers, but in some cases they are based on analyses of the raw mixtures or on the finished cements. The chemical analyses were made so far as possible in accordance with the A.S.T.M. Standard Methods of Sampling and Testing Portland Cement (C 77 – 32).[1]

All the cements were ground to essentially the same fineness as determined by the No. 200 sieve—87.5 per cent passing. Determinations of specific surface by the air analyzer and the Wagner turbidimeter(4) indicated some variation in fineness. The specific surfaces of the 70 cements in Table I by the air analyzer ranged from 1320 to 1690 and averaged 1440 sq. cm. per g. with 57 cements within 5 per cent of the average specific surface and 67 within 10 per cent of the average. The specific surface by the turbidimeter ranged from 1330 to 1960 and averaged 1580 sq. cm. per g. with 43 of the 70 cements within 5 per cent, and 59 cements within 10 per cent of the average. Results of fineness, time of setting, and other miscellaneous tests are given in Table XII.

SCOPE OF TESTS

Specimens were made from the different cements for test as follows:

AT RESEARCH LABORATORY, CHICAGO

1. *Strength Tests:*

(*a*) 3 by 6-in. concrete cylinders of 1:2.4:3.6 mix by weight (water-cement ratio 0.56 by weight) at ages of 28 days, 1, 3½, 5, and 10 years. Cylinders stored in air outdoors and in water in laboratory after 28 days in moist room. Tables II and XI.

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[1] 1933 Book of A.S.T.M. Standards, Part II, p. 6.

(*b*) 2-in. plastic mortar cubes of 1:2.75 and 1:4.25 mixes by weight (water-cement ratios 0.53 and 0.80 by weight, respectively) at ages of 1, 3, 7, and 28 days, 3 months, 1, 2, 3½, 5, and 10 years. Cubes stored in water after 28 days in moist room. Tables III, V, and XI.

(*c*) 1:3 standard sand briquets; ages and storages same as for 2-in. cubes. Tables VII and XI.

(*d*) 3 by 6-in. concrete cylinders, 2-in. cubes and briquets of the same mixes and storages indicated above and exposed to sulfate solutions at age of 28 days. Ages of test for 2-in. cubes and briquets, 3 months, 1, 2, 3½, 5, and 10 years; ages of test for 3 by 6-in. cylinders, 1, 3½, 5, and 10 years. Tables II, III, V, VII, and XI.

2. *Sulfate Exposure:*

3 by 6-in. concrete cylinders, 2-in. plastic mortar cubes and standard sand briquets of the same mixes as indicated above, stored in 2 per cent solutions of sodium sulfate and magnesium sulfate after 28 days in moist room. Tables II, IV, VI, VIII, and XI.

3. *Length Changes:*

(*a*) 3 by 3 by 15-in. Elgin sand mortar bars of 1:3.6 mix by weight, 1 day in sealed mold, then:

(1) 6 days in moist room, then in air of laboratory at 70 F. (21 C.) and 50 per cent relative humidity until 14 months old, then in water for 1 year. Table X.

(2) 89 days in moist room, then in air of laboratory at 70 F. (21 C.) and 50 per cent relative humidity. Table X.

(*b*) 3 by 6-in. concrete cylinders of 1:2.4:3.6 mix by weight stored in water and in 2 per cent solutions of sodium and magnesium sulfate in laboratory after 28 days in moist room. (Fig. 7.)

4. *Accelerated Freezing and Thawing* (*B Cycle*):

3 by 6-in. cylinders of 1:2.4:3.6 mix by weight, cured moist 28 days, then stored in air at 50 per cent relative humidity for 12 to 27 months, then subjected to alternate freezing and thawing. (Fig. 8.)

5. *Miscellaneous Tests.* Table XII:

(*a*) Fineness and surface area.
    (1) Air analyzer.
    (2) Wagner turbidimeter.
(*b*) Water for normal consistency.
(*c*) Soundness.
(*d*) Time of setting.

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1. Fineness, passing No. 200 sieve.
2. Heat evolution(2).
3. Length changes of neat cement specimens in water(1).
4. Length changes of 1:2 standard sand mortar in dry air at about 45 per cent relative humidity and in 2 per cent solutions of sodium and magnesium sulfate(1).

When tests on the laboratory-prepared cements were well under way, several commercial cements were included in the program.

However, the tests on the commercial cements are not far enough advanced to warrant including the results in this report.

In general, three specimens were tested for strength at each age, the individual specimens being made on three different days. Pertinent details of the tests are covered by the notes accompanying the various tables.

INFLUENCE OF CEMENT COMPOSITION ON STRENGTH

Reference to the strength results in Tables II, III, V, and VII, Figs. 1 to 4, and to published tests by other investigators(5–16), reveals that the tensile and compressive strengths of mortar and concrete at various ages of test are markedly influenced by the compound composition of the cement. This is to be expected when it is considered that each compound has its own characteristic strength properties and rate of gain in strength with age, and, in proportion to its amount in the cement, is influencing the strength properties of the cement as a whole. Lack of space does not permit a detailed discussion of the strength results, but a few general indications will be pointed out.

Influence of C_3S and C_2S Compounds on Strength.—The chief strength-giving constituents of portland cement are the two silicates, C_3S and C_2S. In the cements tested, the computed C_3S content ranged from 16 to 74 per cent by weight and the C_2S content from 1 to 58 per cent. The sum of the two silicates ranged from 63 to 84 per cent. With the sum of the other compounds maintained essentially constant, an increase or decrease in the amount of C_3S was accompanied by a concomitant decrease or increase in C_2S. Tests by Bates and Klein(5) and recent tests by the Fellowship(11) on moist-cured specimens of pure C_3S, C_2S, and mixtures of these two silicates have shown the predominating influence of the C_3S on the strength up to 28 days and of the C_2S on the strength beyond the 28-day period. Tricalcium silicate was found to gain its strength quickly while C_2S had relatively little strength at ages up to 28 days, but beyond that age gained in strength at a rapid rate. These findings are borne out by the present tests. Other conditions being the same, the cements high in C_3S (and correspondingly low in C_2S) produced the highest strengths at early ages. However, their *rate of gain* in strength tends to diminish with age so that eventually they may be surpassed in strength by cements low in C_3S (high in C_2S) (see Figs. 1 and 2). These results are based on tests of specimens exposed continuously to moisture. In considering their practical application it is important to note that even under these favorable curing conditions, the cements low in C_3S gained strength slowly. Had the specimens made with such cements been given only short periods of moist curing and then exposed to a dry atmosphere it is probable that they would have shown considerably lower strengths than those reported in the tables, particularly at the later ages.

In the briquet tests (briquets stored 28 days in moist room, then in water), practically all of the cements showed the typical retrogression in strength at the later ages which is characteristic of this

* Cement Double Burned

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 46	56	24	0	16	1	3.2	0.1
No. 6	41	39	0	14	3	3.0	0
No. 45	28	52	0	15	2	3.1	0
No. 42	61	17	7	12	0	3.1	0.2
No. 24	41	37	7	12	0	2.8	0
No. 41	29	49	7	12	0	3.0	0
No. 58*	62	13	14	7	0	3.3	0.7
No. 2B Dupl.	43	32	15	7	0	2.9	0.2
No. 37	16	58	16	6	0	3.0	0.2

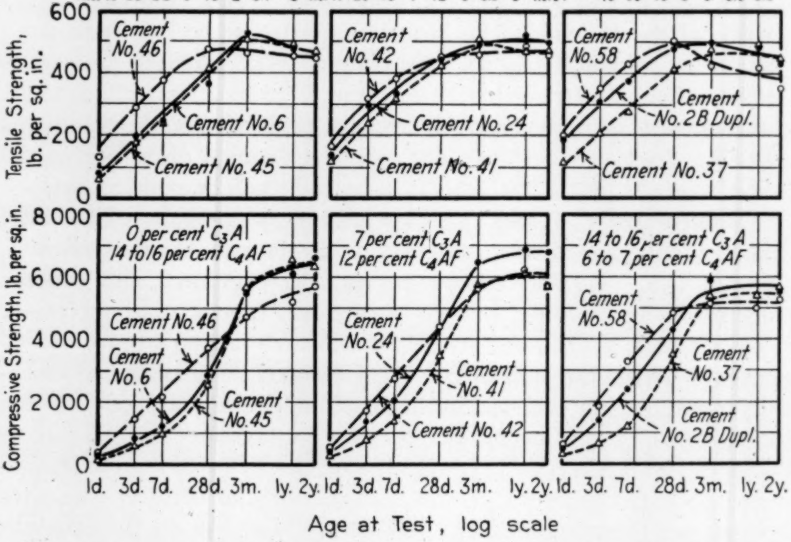
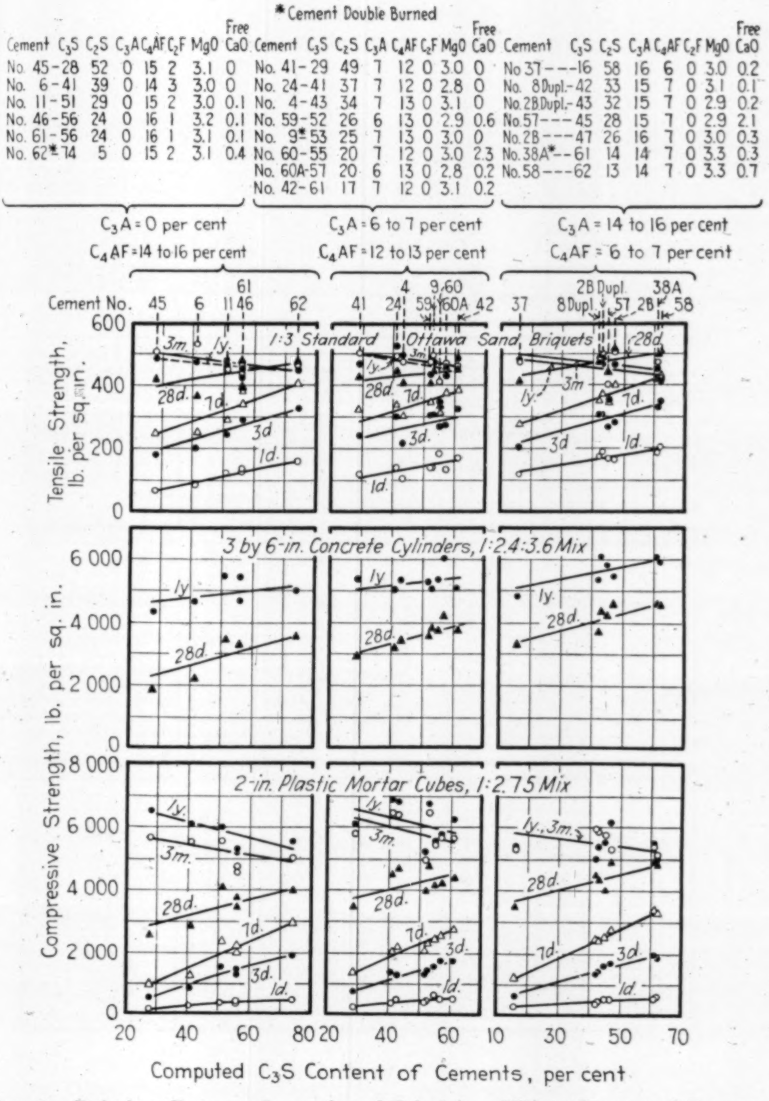


FIG. 1.—Age-Strength Relations for Cements of Varying Tricalcium Silicate Content.

Tension tests made on 1:3 standard Ottawa sand briquets. Compression tests made on 2-in. graded Ottawa sand plastic mortar cubes of 1:2.75 mix by weight. Specimens stored in moist room 28 days, then in water. Data from Tables III and VII.

type of specimen when stored in water. But few of the cements showed any tendency toward retrogression in the compression tests of moist-cured specimens, and where such a tendency occurred, the cements generally contained relatively high amounts of C_3A or C_3A plus C_4AF.

Influence of C_3A and C_4AF Compounds on Strength.—In a study of the effect on strength of increasing the C_3A content, three groups of cements were selected in each of which the C_3A content varied but the C_3S, C_2S, and C_4AF compounds were essentially constant and the



* Cement Double Burned

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 45	28	52	0	15	2	3.1	0
No. 6	41	39	0	14	3	3.0	0
No. 11	51	29	0	15	2	3.0	0.1
No. 46	56	24	0	16	1	3.2	0.1
No. 61	56	24	0	16	1	3.1	0.1
No. 62*	74	5	0	15	2	3.1	0.4

C_3A = 0 per cent

C_4AF = 14 to 16 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 41	29	49	7	12	0	3.0	0
No. 24	41	37	7	12	0	2.8	0
No. 4	43	34	7	13	0	3.1	0
No. 59	52	26	6	13	0	2.9	0.6
No. 9*	53	25	7	13	0	3.0	0
No. 60	55	20	7	12	0	3.0	2.3
No. 60A	57	20	6	13	0	2.8	0.2
No. 42	61	17	7	12	0	3.1	0.2

C_3A = 6 to 7 per cent

C_4AF = 12 to 13 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No 37	16	58	16	6	0	3.0	0.2
No. 8 Dupl.	42	33	15	7	0	3.1	0.1
No. 2B Dupl.	43	32	15	7	0	2.9	0.2
No. 57	45	28	15	7	0	2.9	2.1
No. 2B	47	26	16	7	0	3.0	0.3
No. 38A*	61	14	14	7	0	3.3	0.3
No. 58	62	13	14	7	0	3.3	0.7

C_3A = 14 to 16 per cent

C_4AF = 6 to 7 per cent

Cement No. 45 6 11 61 46 62
41 24 4 59 9 60 60A 42
37 8 Dupl. 2B Dupl. 57 2B 38A 58
1:3 Standard Ottawa Sand Briquets
3m. 1y. 28d. 7d. 3d. 1d.
Tensile Strength, lb. per sq. in.
0
200
400
600
3 by 6-in. Concrete Cylinders, 1:2.4:3.6 Mix
1y. 28d.
2-in. Plastic Mortar Cubes, 1:2.75 Mix
1y. 3m. 28d. 7d. 3d. 1d. 1y., 3m.
Compressive Strength, lb. per sq. in.
0
2 000
4 000
6 000
8 000
20 40 60 80
20 40 60
10 30 50 70
Computed C_3S Content of Cements, per cent

FIG. 2.—Relations Between Strength and Tricalcium Silicate Content of Cement.

Specimens stored in moist room 28 days, then in water. All mixes are by weight. Data from Tables II, III and VII.

*Cement Double Burned

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 50*	47	37	4	8	0	3.0	0.5
No. 16	42	38	9	6	0	2.9	0.9
No. 53*	47	35	10	4	0	3.0	0.3
No. 28	44	34	11	6	0	2.8	0.3
No. 28 Dupl.	43	32	15	7	0	2.9	0.2
No. 57	45	28	15	7	0	2.9	2.1
No. 8 Dupl.	42	33	15	7	0	3.1	0.1
No. 2B	47	26	16	7	0	3.0	0.3
No. 1	43	27	18	4	0	2.8	1.4
No. 1 Dupl.	45	28	20	4	0	2.9	0.2

C_4AF = 4 to 8 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 6	41	39	0	14	3	3.0	0
No. 51	38	45	2	13	0	3.3	0
No. 26	42	38	3	15	0	2.7	0
No. 4	43	34	7	13	0	3.1	0
No. 24	41	37	7	12	0	2.8	0
No. 29	40	33	11	13	0	2.8	0
No. 12	41	32	12	12	0	2.8	0.1
No. 20	42	30	12	13	0	2.8	0
No. 30	39	32	13	13	0	2.8	0.1

C_4AF = 12 to 15 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 19	44	33	0	19	1	2.5	0
No. 27	41	35	1	20	0	2.9	0
No. 17	39	37	3	18	0	2.9	0
No. 25	44	32	4	18	0	2.5	0
No. 23	38	33	5	20	0	2.8	0
No. 13	38	31	7	21	0	2.7	0
No. 21	40	30	10	17	0	2.8	0

C_4AF = 17 to 21 per cent

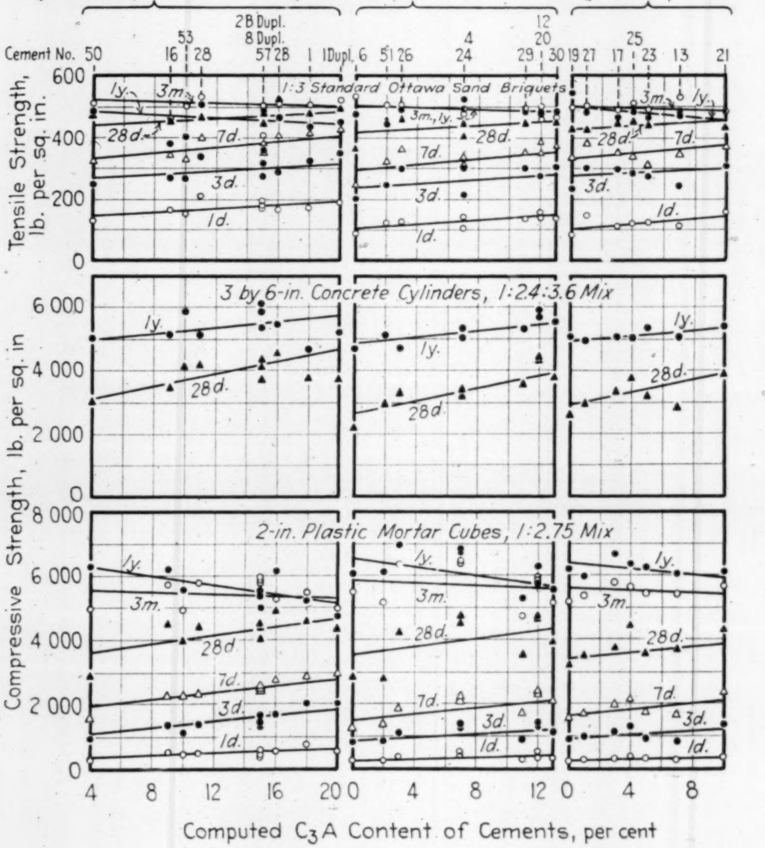


FIG. 3.—Relations Between Strength and Tricalcium Aluminate Content of Cement.

Specimens stored in moist room 28 days, then in water. All mixes are by weight. Data from Tables II, III and VII.

*Cement Double Burned

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 7	42	39	0	10	6	3.0	0
No. 6	41	39	0	14	3	3.0	0
No. 5B	39	40	2	16	0	3.1	0.1
No. 19	44	33	0	19	1	2.5	0
No. 27	41	35	1	20	0	2.9	0

C_3A = 0 to 2 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 50*	47	37	4	8	0	3.0	0.5
No. 18	43	39	5	9	0	2.8	0.4
No. 24	41	37	7	12	0	2.8	0
No. 4	43	34	7	13	0	3.1	0
No. 14	37	38	7	15	0	2.8	0
No. 22	41	33	8	15	0	2.9	0
No. 25	44	32	4	18	0	2.5	0
No. 23	38	33	5	20	0	2.8	0
No. 13	38	31	7	21	0	2.7	0

C_3A = 4 to 8 per cent

Cement	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	Free CaO
No. 53*	47	35	10	4	0	3.0	0.3
No. 28	44	34	11	6	0	2.8	0.3
No. 34	41	36	11	9	0	1.4	0.2
No. 35	41	33	11	9	0	4.9	0.2
No 3B No. 3B Dupl.	43	33	11	10	0	3.1	0.3
No. 12	41	32	12	12	0	2.8	0.1
No. 20	42	30	12	13	0	2.8	0
No. 21	40	30	10	17	0	2.8	0

C_3A = 10 to 12 per cent

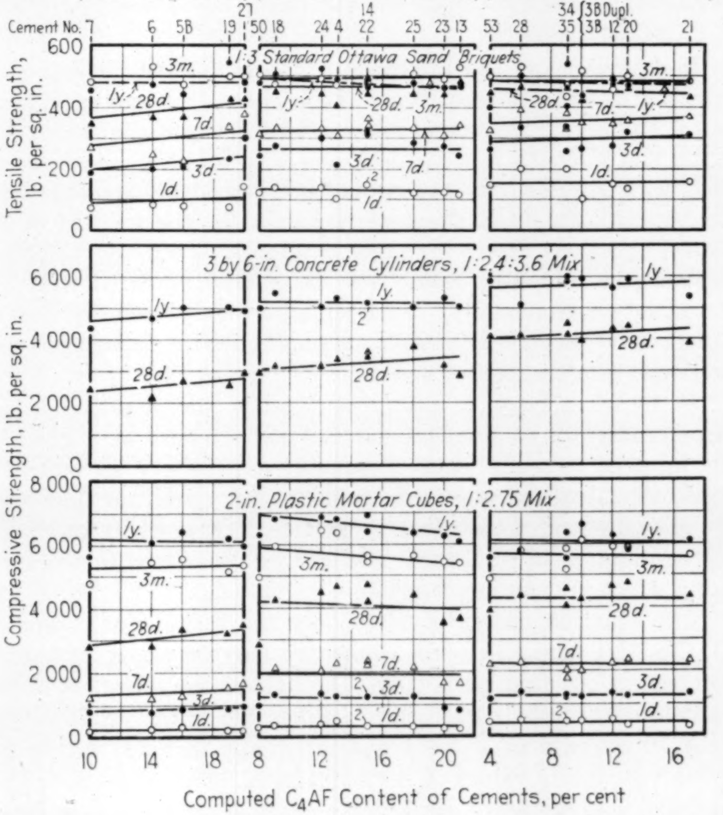


FIG. 4.—Relations Between Strength and Tetracalcium Alumino Ferrite Content of Cement.

Specimens stored in moist room 28 days, then in water. All mixes are by weight. Data from Tables II, III and VII.

C_4AF varied from group to group (Fig. 3). At ages up to 28 days both the compressive and tensile strengths increased with increase in the C_3A content, but at later ages there was, in general, a tendency toward lower strengths for the higher C_3A contents, other conditions being the same.

Results of tests on three groups of cements each containing approximately constant amounts of C_3S, C_2S, and C_3A but variable amounts of C_4AF indicate that the C_4AF compound produced little, if any, change in strength (Fig. 4).

Mathematical Study of Influence of Cement Compounds on Strength (Moist Curing):

In investigating the influence of the individual compounds on the strength of the different types of specimen at various ages, mathematical studies of the general relationships between the cement compounds and strength were made in order to find a type of equation which, with suitable constants, would give reasonably good agreement between calculated and observed values. By the method of least squares, equations expressing these relationships were developed and the strengths computed from these equations compared with those determined experimentally. Such equations are necessarily empirical as there is no theoretical basis for assuming that a particular type of equation is fundamentally correct.

In these analyses, a linear relationship[1] was assumed between the percentage of each compound and the strength at a given age. This form of equation gives reasonably satisfactory agreement between calculated and observed strengths but could not be fitted to the data on some of the other properties. Other types of equation may be required in such cases and will be considered in the further studies which are contemplated.

The equation chosen involves only the four major compounds, C_3S, C_2S, C_3A, and C_4AF. A compound may be neglected only if it is present in constant proportions throughout the series, or if its influence on strength is negligible. Upon the latter assumption, all of the 70 cements in Table I were originally included in the series used in calculating values of the coefficients for the equations by the method of least squares. However, a study of the differences between

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[1] The linear equation used to express the general relation between composition and strength, for a given type of specimen and age at test, is of the usual form,

$$a\ C_3S + b\ C_2S + c\ C_3A + d\ C_4AF = \text{Strength}$$

A constant term is not introduced, since any linear equation with a constant term may be converted to the above form through the relation,

$$C_3S + C_2S + C_3A + C_4AF = k$$

where k is the sum of the percentages of the four compounds. In this series, since the sum is practically constant, there is no advantage in introducing a constant term.

calculated and observed strengths showed that those in poorest agreement differed from the greater portion of the series either in free CaO or MgO content, or both, indicating that the influence of these components was not negligible. The number of such cements is too small to justify adding terms to the equations for the purpose of including the influence of MgO and free CaO. Therefore, cements with more than 1 per cent free CaO, or with less than 2.5 and more than 3.4 per cent MgO were omitted in the mathematical analysis of the strength data. In calculating the effect of C_4AF compound, the C_2F, where present, was included with the C_4AF since the C_2F is in solid solution with C_4AF as a single solid phase.

The coefficients obtained, with their probable errors, are given in Table IX. The fact that a linear equation fits the data reasonably well indicates that within the range of compositions included in the mathematical analysis an additive effect of the compounds may be assumed, subject to the calculated probable errors. The coefficients may, therefore, be used as a basis for studying the influence of each compound with time for each type of specimen, since they show the progressive effect of each compound up to 2 years for the particular conditions of these tests.

Inspection of the coefficients obtained by this method of analysis shows that for all types of specimen the C_3S is continuously effective in increasing strength. The C_2S contributes little to strength up to about 28 days but is largely responsible for the increases in strength beyond that period.

At the 7- and 28-day periods for the cubes and at all ages up to 28 days for the briquets, the C_3A appears to exert a greater effect on strength than any of the other constituents. Its effect on strength diminishes beyond the 28-day period and at 1 and 2 years is either nearly zero or negative. In the case of the concrete cylinders the decrease in the effectiveness of the C_3A after the 28-day period was less rapid.

The C_4AF had a negative effect on the compressive strength of the 1:2.75 cubes up to about 7 days, and in the case of the 1:4.25 cubes its effect was slight at all ages. In the case of the briquets the C_4AF appears to add to the strength after 1 day, although its effect diminishes beyond the 3-month period.

Davis, Carlson, Troxell, and Kelly report strength factors at 7, 28, and 90 days for compression tests of moist-cured 2 by 4-in. mortar cylinders of approximately 0.57 water-cement ratio by weight(10). The cylinders were made with Boulder Dam sand using 20 cements of equal fineness. While the factors they obtained differ in degree from those in Table IX at corresponding ages, except for the C_4AF compound they indicate the same general trends.

The high values of the C_3A factors at 28 days are not easily explained. Bogue and Lerch consider the effect to be due to a secondary cause rather than to a direct contribution to strength of the order indicated. They suggest that since C_3A requires a larger amount of water for hydration than the other cement compounds, increasing its percentage in a series of tests such as this where a nearly uniform quantity of water is used for mixing, will increase the amount of water which will be taken up by the C_3A leaving decreasing amounts for the silicates. The resulting decreased water-silicates ratio may then result in the improved strengths commonly associated with decreased water-cement ratios(11). Bogue and Lerch further suggest that the negative values of the factors for C_4AF may be due to a deposition of colloidal, hydrated $CaO \cdot Fe_2O_3$ around the grains of the other compounds, thus delaying their rate of reaction with water(11).

INFLUENCE OF CEMENT COMPOSITION ON LENGTH CHANGES OF MORTAR BARS

Length Changes of Mortar Bars.—Table X contains the measured expansions and contractions of 3 by 3 by 15-in. mortar bars of 1:3.6 mix by weight when stored under different conditions. The data for the nine groups of cements used in the strength studies of Figs. 2 to 4 were examined separately in order to determine whether any significant trends existed between the measured length changes of the bars and each of the four principal cement compounds. Expansion and contraction were found to be virtually independent of the relative proportions of C_3S, C_2S, and C_4AF, but variations in the proportion of C_3A had an appreciable influence. In general, as the C_3A content increased there was a tendency toward increased expansion of the bars during their 3-month storage in the moist room, and increased total contraction after 14 months and 2 years storage for bars allowed to dry in the air of the laboratory at about 50 per cent relative humidity. The tests included both 7 days and 3 months storage in moist room before drying (Fig. 5). For the bars moist-cured for 7 days, the values of total contraction at 14 months ranged from about 0.07 per cent for a C_3A content of 0 per cent, to about 0.11 per cent for a C_3A content of 20 per cent. Approximately the same range in total contraction was obtained at age of 2 years for the bars which were moist cured for 3 months before they were allowed to dry out in air of laboratory.

Relationship Between Composition and Total Contraction.—Linear equations were found to represent relations between composition and length changes of the bars with reasonably satisfactory agreement between observed and calculated values. The equations were derived from the same 57 cements used in studying the strength data. It will be recalled that these cements contained 1 per cent or less of free CaO and approximately 3 per cent of MgO. The coefficients and their probable errors, obtained by the method of least squares, were as follows:

Compound	Coefficients of Linear Equations Representing Relation Between Composition and Total Contraction of Mortar Bars, per cent	
	After 7 days moist curing, Total Age 12 months	After 3 months moist curing, Total Age 15 months
C_3S	0.00079 ± 0.000036	0.00082 ± 0.000036
C_2S	0.00077 ± 0.000033	0.00077 ± 0.000035
C_3A	0.00234 ± 0.000100	0.00213 ± 0.000105
C_4AF	0.00049 ± 0.000114	0.00046 ± 0.000118

The coefficients for $11\frac{3}{4}$ months storage in air after 7 days in moist room do not differ greatly from the values for 12 months storage in air following 3 months storage in moist room. The two silicates appear to have influenced the total contraction to approximately the same degree but to a much lesser degree than the C_3A and to a somewhat greater degree than the C_4AF compound. Woods, Steinour, and Starke(17) and Bogue and Lerch(1) also report that the C_3A compound has the greatest influence on the total contraction of mortar bars.

INFLUENCE OF CEMENT COMPOSITION ON RESISTANCE TO SULFATE SOLUTIONS

Valuable information on the behavior of portland cement when exposed to the action of sulfates has appeared in the literature, particularly in recent years(9,18–27). These tests have developed considerable additional information on this subject and supplement the results presented in the recent report by Bogue and Lerch(1).

Ratings and Durability Factors.—Concrete cylinders, 2-in. plastic mortar cubes and standard briquets made with each cement were, after 28 days of moist curing, stored in large tanks containing 2 per cent solutions of magnesium and sodium sulfate. The solutions were kept essentially constant by periodic adjustments of their sulfate content.

The influence of composition on the resistance of the cements to the solutions is clearly shown by the ratings and strengths of the specimens (Tables II to VIII) after various periods of exposure. The ratings were determined by visual inspection. The durability factors at the 1, 2, and 3-year periods recorded in the tables were devised to facilitate the study and plotting of the results. These factors at a given yearly period are the sums of the separate ratings at all preceding 6-month periods. For example, the concrete specimens for cement No. 1 Duplicate, Table II, in the magnesium sulfate solution have ratings of 9.0 and 8.0 at 6 months and 1 year, respectively. The sum of these two ratings, 17.0, is the durability factor at the 1-year period. The ratings at 1½ and 2 years are 6.6 and 2.6. These ratings added to those at 6 months and 1 year give the durability factor at the 2-year period, 26.2. Similarly, the ratings of 0.3 and 0 at 2½ and 3 years added to those at previous 6-month periods give

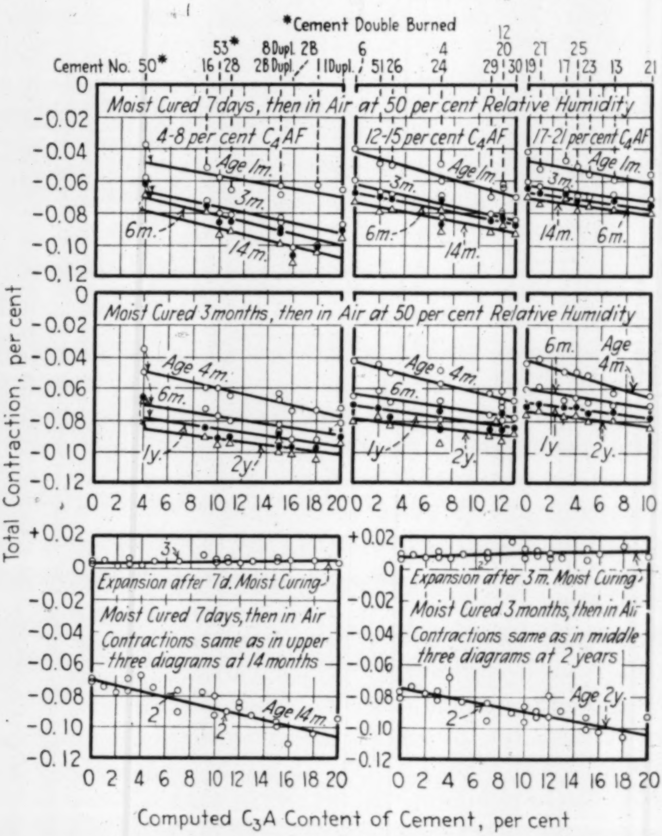

FIG. 5.—Length Changes of Mortar Bars as Influenced by Tricalcium Aluminate Content of Cements.

Data from Table X. Compare with Fig. 3. Specimens: 3 by 3 by 15-in. mortar bars, 1:3.6 mix by weight. Aggregate: Elgin sand graded 0 to No. 4. Numerals at plotted points indicate number of identical values.

the durability factor at the 3-year period of 26.5. Durability factors for specimens in apparently sound condition at the end of 1 year are 20, at 2 years, 40, and at 3 years, 60 (a rating of 10 for each 6-month period). These latter values are the maximum attainable and values between them and zero represent intermediate degrees of resistance at the particular period considered. By this method the behavior of the specimens during a given period can be represented by a single factor which to some extent indicates the rate at which disintegration has occurred.

Resistance of Cements to Sulfate Solutions.—The data on the resistance of the cements to the sulfate solutions as influenced by each

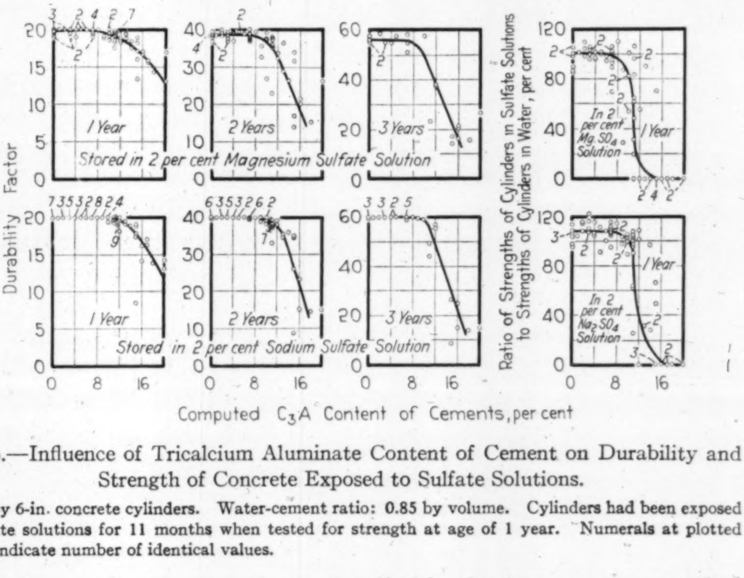

FIG. 6.—Influence of Tricalcium Aluminate Content of Cement on Durability and Strength of Concrete Exposed to Sulfate Solutions.

3 by 6-in. concrete cylinders. Water-cement ratio: 0.85 by volume. Cylinders had been exposed to sulfate solutions for 11 months when tested for strength at age of 1 year. Numerals at plotted points indicate number of identical values.

of the four major compounds was studied in the same manner as that employed in Figs. 2 to 5 in studying strength and length changes. No significant trends were observed for any of the compounds except C_3A. These studies as well as analyses of the durability factors showed that the C_3A had a marked detrimental influence on the sulfate resistance of the cements.

The rate of disintegration varied somewhat with the type of specimen and the solution. It was probably influenced more by the difference in physical characteristics of the paste than by the shape of the specimens. The type of aggregate was probably a factor as well.

The concrete cylinders appeared to disintegrate more slowly than the other types of specimen in both solutions. Good resistance up to the 3-year period was exhibited by the concrete cylinders when the C_3A content of the cement was 7 per cent or less in the case of the cylinders stored in magnesium sulfate solution, and when 10 per cent or less in the case of the cylinders stored in sodium sulfate solution (Fig. 6). Where the C_3A content was greater than these values the extent of disintegration at the 3-year period increased with increase in the amount of C_3A present.

In general, the cubes and briquets made with cements containing 11 per cent or more of C_3A showed marked attack at 1 year in both solutions, but in the case of the concrete specimens only those made with cements containing 15 per cent or more of C_3A showed considerable attack at this period. It is of particular interest to note that the lean 1:4.25 cubes made with cements low in C_3A showed much better performance than the richer 1:2.75 cubes made with cements high in C_3A. This confirms the findings of Bogue and Lerch that certain changes in composition have a greater effect upon resistance to sulfate attack than changes in richness of mix and water-cement ratio(1). In this connection it should be pointed out that the resistance of the richer mixtures was in practically all cases greater than that of the lean mixture of the same cement, showing the importance of control of the quality of the concrete where exposure to sulfate waters is to be expected.

From diagrams showing durability factors plotted against C_3A contents, similar to those in Fig. 6, the maximum C_3A contents for good resistance to the solutions were determined for each type of specimen. The values follow:

Type of Specimen	Water-Cement Ratio, by weight	Maximum C_3A Contents for Good Sulfate Resistance at Period Indicated					
		2 per cent $MgSO_4$ Solution			2 per cent Na_2SO_4 Solution		
		1 year	2 years	3 years	1 year	2 years	3 years
3 by 6-in. concrete cylinders	0.563	10	8	7	12	11	10
1:2.75 mortar cubes	0.533	8	7	6	9	8	6
1:4.25 mortar cubes	0.800	8	8	7	8	7	6
1:3 briquets	0.400	9	8	6	12	10	9

It is seen from the above values for the 3-year period that good resistance to the magnesium sulfate solution was obtained with all types of specimen when the C_3A content did not exceed 6 or 7 per cent, and in the case of the sodium sulfate solution when it did not exceed 6 to 10 per cent.

Strength of Specimens Stored in Sulfate Solutions.—The strength of specimens stored in the sulfate solutions expressed as a percentage of the strength of similar specimens stored in water and tested at the same age was used to determine whether any impairment in strength occurred during storage in the solutions. In these comparisons the specimens when tested were of the same total age. However, those exposed to the solutions had been in the solution one month less than the age at test since they were moist cured one month before exposure to sulfate action.

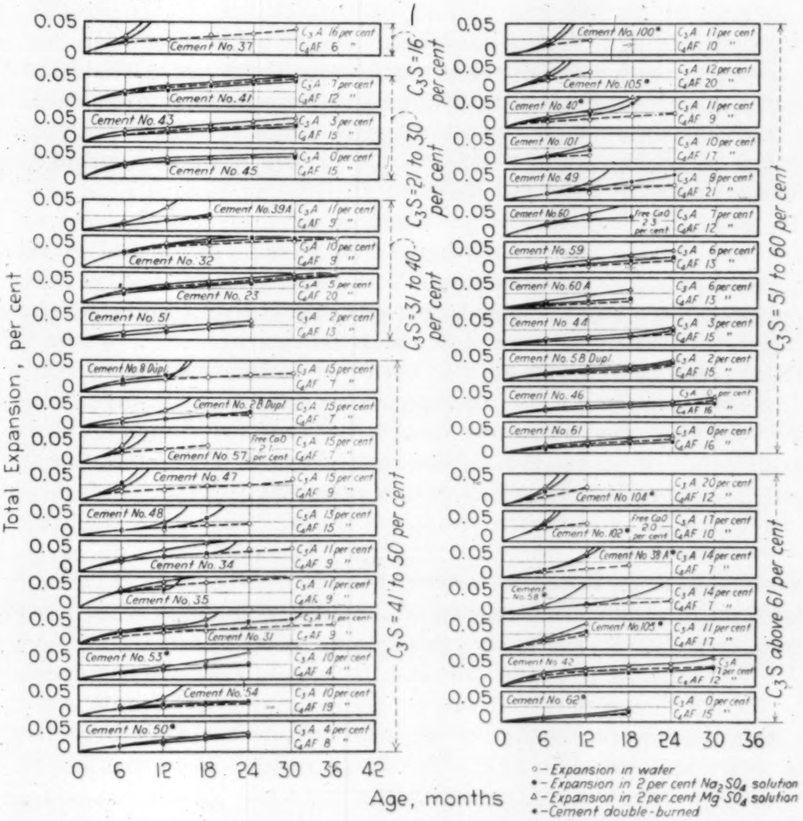

FIG. 7.—Expansion of 3 by 6-in. Concrete Cylinders in Water and in Sulfate Solutions.

Specimens moist cured 28 days before storage in water and in sulfate solutions.

When the specimens exposed to the solutions had begun to disintegrate some difficulty was encountered in testing them for strength. This was particularly true of the compression specimens and some had to be capped before loading due to poor condition of the bearing surfaces as a result of the disintegration and swelling which had occurred. In most cases where the ends and edges of the compression specimens were badly affected, no attempt was made to test them and they were considered as having disintegrated completely although the portion of the specimen that remained still possessed considerable strength. All strength values in the tables are based on the original cross-sections of the specimens.

The strength ratios for each type of specimen and age of test were plotted in the same manner as that shown for the concrete cylinders in Fig. 6. From these diagrams the approximate C_3A contents for strength ratios of 100 per cent were determined. The values obtained in this manner were, at corresponding ages, in fair agreement with the maximum C_3A contents for good sulfate resistance discussed in the preceding section which were derived from the durability factors. The principal difference occurred for the briquets stored in the magnesium sulfate solution where a value of zero per cent for C_3A was obtained on the strength ratio basis at the 1 and 2-year periods. The briquet strength ratios for zero per cent C_3A at these two periods averaged 93 and 70 per cent, respectively, and showed a fairly regular decrease from these values to a strength ratio of zero at a C_3A content of about 12 per cent. It is believed that the low strengths of the briquets in the magnesium sulfate solution are due to the relatively large reduction in effective area for a comparatively small depth of penetration of the sulfate action. A penetration of only 0.05 in. reduces the effective area about 20 per cent.

Expansion of Concrete Cylinders in Sulfate Solutions.—The linear expansions of 3 by 6-in. concrete cylinders made from 38 of the cements were measured at various periods of storage in 2-per-cent solutions of sodium and magnesium sulfate and in water after 1 day in mold and 27 days in moist room.

The expansions of the cylinders in the solutions conform closely to those of the cylinders in water until the action of the sulfate causes a sharp increase in the rate of expansion which marks the beginning of visual disintegration (Fig. 7). In general, as the expansion increases the extent of disintegration also increases. There is a noticeable decrease in the expansion of the cylinders stored in the solutions with decrease in C_3A content of the cement. Where the C_3A content is 7 per cent or less the expansions up to the 2-year period are not excessive except in the case of cement No. 60 which contained 2.3 per cent uncombined lime. The expansions in water also tended to decrease with decrease in C_3A content.

INFLUENCE OF CEMENT COMPOSITION ON RESISTANCE TO FREEZING AND THAWING

When this investigation was started, freezing-and-thawing tests were not included in the program. However, as the tests progressed it seemed desirable to obtain some preliminary information on the resistance to alternate freezing and thawing of concrete made with the various cements. There were available for this purpose three 3 by 6-in. concrete cylinders each for 66 of the cements listed in Tables I and XI. These cylinders had been moist cured 28 days and then stored in air of laboratory at approximately 50 per cent relative humidity for periods of 12 to 27 months. Many of the cylinders had stainless steel gage points embedded at the ends and had been measured at definite intervals for length changes during the period of dry-air storage. A sufficient number of the remaining cylinders were provided with gage points set in neat cement paste in holes drilled in the ends so that length changes occurring during the freezing-and-thawing cycles could be observed on at least 2 specimens for each cement. Losses in weight were obtained on 3 specimens for each cement.

All cylinders were weighed and then soaked in water for 5 days before being subjected to the alternate freezing-and-thawing cycles. The absorptions occurring during the soaking period were recorded.

Freezing-and-Thawing Apparatus.—This series of tests was carried out using a different technique from any previously reported by this laboratory(31,33,34). The apparatus consists of an insulated concrete tank with four compartments each filled with calcium chloride brine. The brine is cooled by coils of pipes through which ammonia is circulated. The 3 by 6-in. cylinders were completely immersed in water in metal containers, 6¼ by 6¼ in. in cross-section and 32 in. high, which were placed directly in the brine. The spaces in the corners of the rectangular tank and in the center between the cylindrical specimens were partially filled with cast-iron bars of triangular cross-section so that the amount of water required to surround and completely immerse the specimens was reduced to a minimum. Circulation of the brine in each compartment was obtained by means of a motor-driven agitator.

The capacity of the apparatus is such that approximately one hundred and fifty 3 by 6-in. cylinders could be cooled simultaneously from 50 F. (10 C.) to +10 to −5 F. (−12 to −20.5 C.) in a period of 1½ hr. The brine temperature ranged from 0 to −15 F. (−18 to −26 C.).

"B" Cycle of Tests.—For the tests reported herein, the cycle of operations, termed the *B* cycle was as follows: The containers holding the specimens were allowed to remain in the agitated cold brine 1½ hr., after which they were placed in a tank containing warm water for a period of from 1 to 1½ hr. During this period the specimens and surrounding water remained in the containers. The temperature and

quantity of water in the tank were adjusted so that the ice and specimens within the containers were completely thawed and raised to a temperature of about 50 F. (10 C.) in the 1 to 1½-hr. period. Circulation of the water in the thawing tank was maintained by means of a motor-driven agitator.

In one of the containers in each of the compartments at least two "dummy" specimens were included in which thermocouples had been embedded. Readings were taken at regular intervals to indicate the temperature of the specimens at the bottom and top of the container. Also a thermocouple was submerged in the brine of each tank to record the temperature of the calcium chloride.

The regular routine followed in this *B* cycle was to freeze and thaw three times daily. After the last thawing, the specimens remained in the containers in the thawing tank until the following morning when they were again placed in the brine tanks and the three daily cycles repeated. After 4 days, or 12 of these cycles, the specimens were removed from the containers, weighed and measured for change in length. They were then placed in a gas-fired drying oven at 120 F. (49 C.) for a total of 72 hr. After removal from the oven the specimens were allowed to cool at room temperature for a period of about 16 hr., the weight and length were again recorded, and the specimens placed in water. After remaining in water for 72 hr. they were weighed, measured for length and then replaced in the containers for the beginning of the next series of freezing-and-thawing cycles. In replacing the cylinders after each 12 cycles their position in the containers was rotated in a systematic manner so that each received as nearly as possible the same treatment.

The 12 cycles of freezing and thawing, the 72-hr. drying and 72-hr. saturation periods, plus the time for cooling and measuring required two weeks. By dividing the specimens into two groups, one set received the freezing-and-thawing cycle while the other set was receiving the drying and saturation treatment. During the freezing each group was again divided so that one-half of the specimens was thawing while the other half was freezing.

Length Changes and Losses in Weight of Test Cylinders.—Typical curves illustrating the expansion and loss in weight resulting from the indicated number of *B* cycles of freezing and thawing are shown in the upper two diagrams of Fig. 8. The expansions and losses in weights shown are those determined at the time the specimens were removed from the thawing tank on the morning following each twelfth cycle and before introduction into the oven. The figures recorded refer only to the number of freezings and thawings. For each 12 cycles recorded there was an accompanying cycle of drying and saturation as described previously. In a few instances, due to mechanical difficulties with the compressor, the drying cycle was introduced following 9 or 10 cycles of freezing and thawing. In the total period represented by 300 cycles of freezing and thawing, this variation in procedure occurred on only 7 occasions.

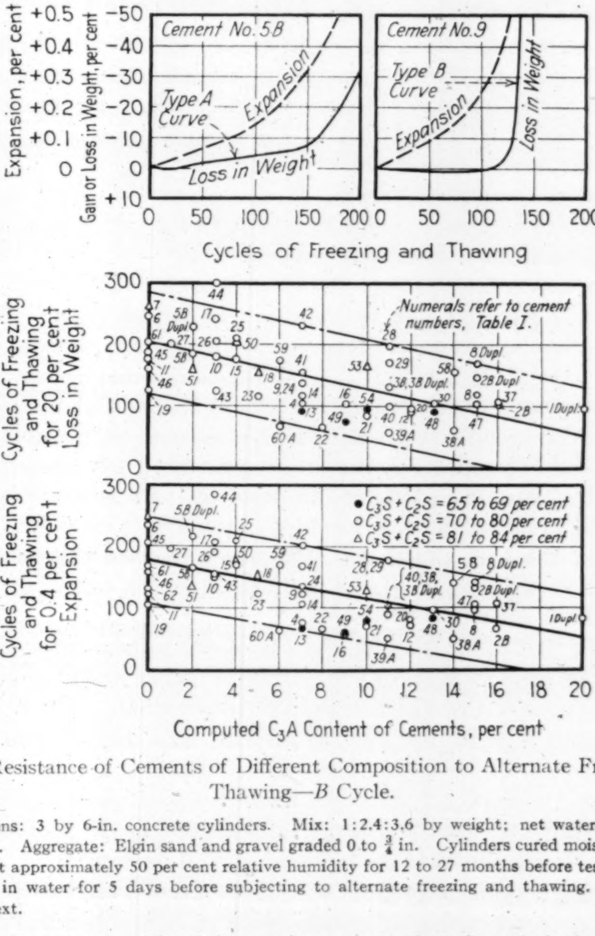

FIG. 8.—Resistance of Cements of Different Composition to Alternate Freezing and Thawing—*B* Cycle.

Specimens: 3 by 6-in. concrete cylinders. Mix: 1:2.4:3.6 by weight; net water content 6.35 gal. per sack. Aggregate: Elgin sand and gravel graded 0 to $\frac{3}{4}$ in. Cylinders cured moist for 28 days, then in air at approximately 50 per cent relative humidity for 12 to 27 months before test. All cylinders soaked in water for 5 days before subjecting to alternate freezing and thawing. For further details see text.

Of 66 cements tested for resistance to freezing and thawing, 42 showed loss-in-weight curves similiar to the type *A* curve while for the remaining 24 cements the loss-in-weight curves were similar to the type *B* curve (Fig. 8). With the type *A* curve there occurred a relatively slow and gradual attrition of the specimens under the freezing-and-thawing treatment. With the type *B* curve, little if any loss and in some cases a small gain in weight was indicated as the number of cycles increased. This continued until a very marked loss occurred and total failure resulted after a few additional cycles. The type *A* curve occurred most frequently in cements relatively low in C_3A and the type *B* in cements relatively high in C_3A. Occasionally, however, the type *A* curve was obtained with cements relatively high in C_3A and the type *B* with cements low in C_3A.

It is to be noted that for both types of loss-in-weight curves, the expansions of the cylinders gradually increased with increase in the number of cycles. A sharp increase in loss in weight generally occurred when the measured expansions of the cylinders amounted to 0.3 to 0.5 per cent in the case of the type *A* curves and 0.4 to 0.6 per cent in the case of the type *B* curves.

Resistance to Freezing and Thawing as Influenced by Cement Composition.—Numerous studies were made of individual groups of cements to see whether there was any variation in their resistance to alternate freezing and thawing due to variation in composition. Statistical studies of the data were also made. On the basis of the loss in weight and expansion of the specimens, these various studies indicated a trend toward increased resistance with increase in the C_3S and C_2S compounds and a trend toward decreased resistance with increase in the C_3A and C_4AF compounds, the latter compound having considerably less effect than C_3A. Hughes, in tests on 14 commercial portland cements in a mortar mix of 0.70 water-cement ratio by weight, likewise found that the C_3A and C_4AF compounds reduced the resistance to freezing and thawing in the *S* cycle used in his tests but that the C_4AF had the greater influence (27).

In the two lower diagrams of Fig. 8 the number of cycles which produced an expansion of 0.4 per cent and a loss in weight of 20 per cent is plotted against the C_3A content of the cements. The numbers in the diagrams identify the various cements and the different symbols serve to indicate their approximate total silicate contents. While the plotted values show considerable spread in both diagrams a definite trend toward increased resistance is indicated as the C_3A content is reduced. It appears that the influence of C_3A on resistance to freezing and thawing is sufficiently great so as not to be masked by the influence of the other compounds. It is probable that several factors such as the presence of chert particles in the coarse aggregate, the position of the specimens in the containers, differences in the degree of hydration and in age at time of test, and the limited number of specimens of each kind, contributed to some extent in producing the spread indicated in the diagrams. Further statistical studies of the data may serve to indicate the influence of composition with respect to each of the compounds involved. In addition, an extension of this series is now under way, with improved control in the freezing-and-thawing test, for the purpose of checking these results and eliminating that portion of the spread which may be caused by variations in testing procedure.

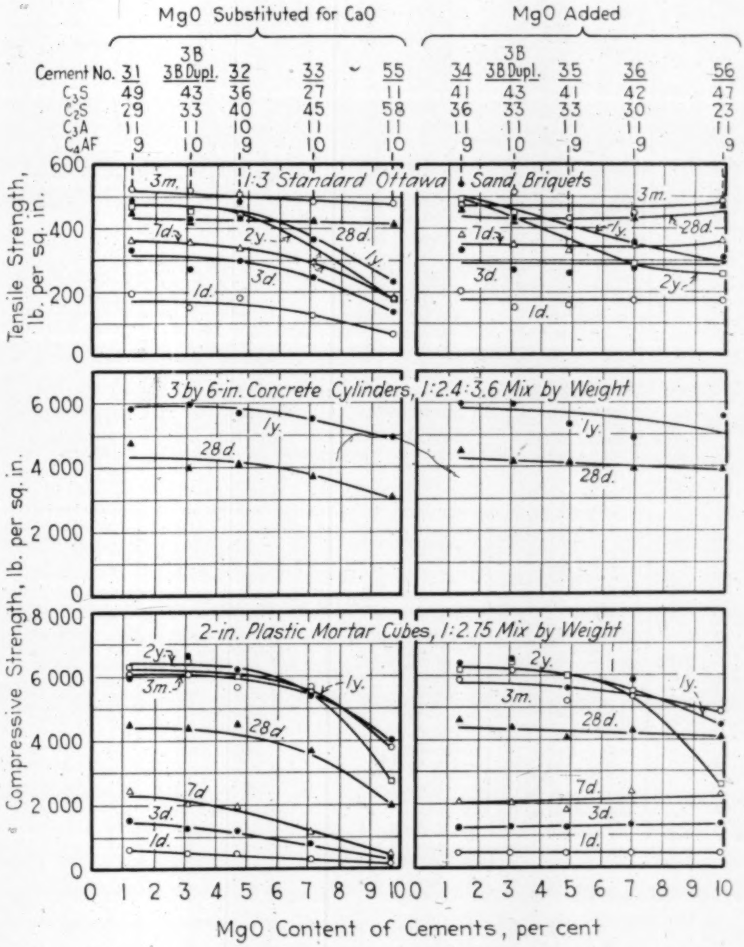

FIG. 9.—Strength of Cements of Varying MgO Content.
Data from Table XI. Specimens stored in moist room 28 days, then in water.

TESTS OF CEMENTS WITH VARYING MGO CONTENT

In a group of 12 cements the composition was varied in two ways by varying the MgO content. In 6 of the cements (Table XI) MgO was substituted for CaO in the raw mix causing the C_3S to decrease from 49 to 11 per cent and the C_2S content to increase from 29 to 58 per cent while the C_3A and C_4AF remained substantially constant at 11 and 10 per cent respectively. The MgO content of these 6 cements varied from 1.2 to 9.7 per cent.

In the other 6 cements the MgO was used as an addition to the raw mix. As the MgO was introduced into the mix, adjustments were made in the CaO and SiO_2 to maintain the C_3S approximately constant at 45 per cent. The C_2S decreased from 36 to 23 per cent and the C_3A and C_4AF remained constant at 11 and 9 per cent respectively. The MgO in these cements varied from 1.4 to 9.9 per cent.

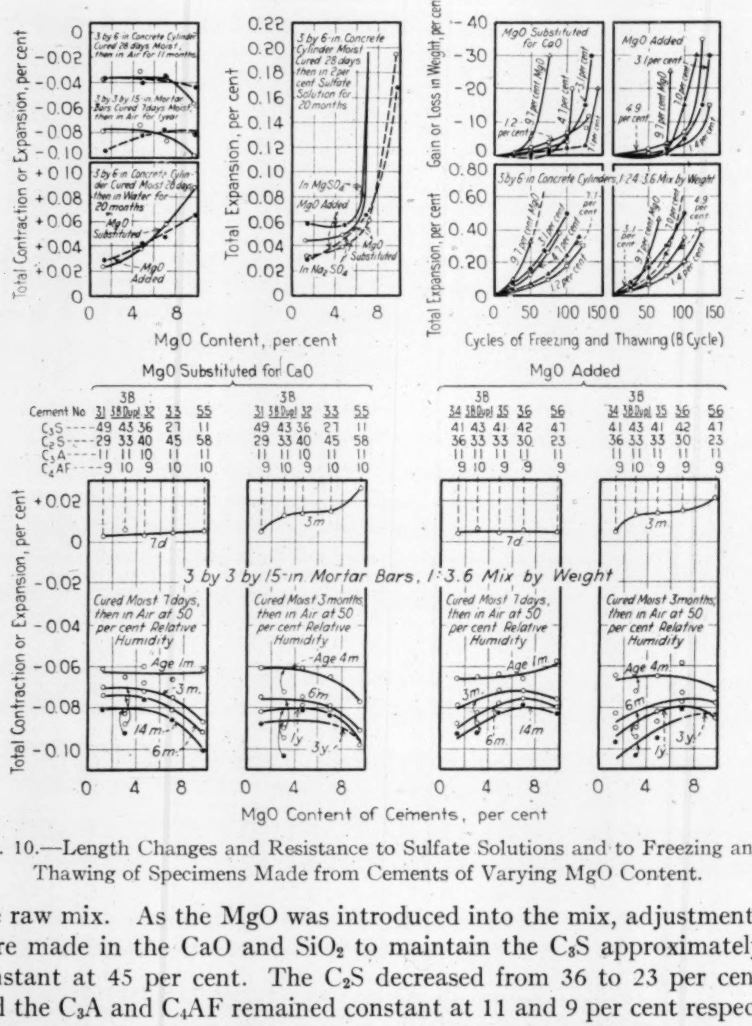

FIG. 10.—Length Changes and Resistance to Sulfate Solutions and to Freezing and Thawing of Specimens Made from Cements of Varying MgO Content.

Influence of Varying MgO Content on Strength.—When MgO was *substituted for CaO* there was a gradual decrease in strength with increase in MgO content at all ages (Fig. 9). This apparent reduction in strength is largely due to the change in the relative percentages of the 2 silicates and probably to some extent to the reduction in total silicates with increase in MgO content. This will be evident if the strengths for these 6 cements are compared with the strengths of other cements (from Table I) containing similar percentages of the C_3S, C_2S, C_3A and C_4AF compounds. Such a comparison reveals little, if any, difference in strength except in the case of cements Nos. 33 and 55 which contained 7.1 per cent and 9.7 per cent MgO respectively. Cement No. 33 showed reduced briquet strengths at the 1- and 2-year periods. Cement No. 55 generally showed lower strengths at all ages than cement of approximately similar composition except for MgO content. The low strengths shown by the cubes and briquets at the 2-year period for cement No. 55 are particularly noteworthy.

The *addition* of MgO up to 9.9 per cent with the C_3S, C_3A, and C_4AF kept essentially constant had little effect on strength of either mortars or concrete except at 1 and 2 years when there was a decrease for the two highest percentages. This decrease was most noticeable at the 2-year period and was nearly as pronounced as in the group where MgO was substituted for CaO. In this case also consideration should be given to the reduction in total silicates as a partial cause for the loss in strength. The effect of MgO on strength has been reported by Bates(36) and more recently by Kühl(39). The results obtained by these investigators are in general agreement with those reported here where the conditions of test are comparable.

Influence of MgO on Length Changes of Mortar and Concrete.—Recent information indicates that the MgO present in normal portland cement is uncombined with other compounds(38). The hydration of this MgO proceeds slowly with time and with considerable increase in volume(37). This being the case it is of interest to investigate the behavior of the specimens containing cements high in MgO when stored in water, in air, and in sulfate solutions.

Concrete cylinders stored in 2-per-cent solutions of magnesium and sodium sulfate up to 20 months exhibited excessive expansion followed in some cases by complete disintegration where the MgO content of the cement was greater than 5 or 6 per cent in the case of cylinders stored in magnesium sulfate, and when greater than 7 per cent in the case of cylinders stored in sodium sulfate. In the latter solution, cement containing 7 per cent MgO showed about 50 per cent greater expansion than cement containing 5 per cent MgO.

Concrete cylinders stored in water for 20 months showed increased expansion with increase in MgO content of the cement (Fig. 10).

Concrete and mortar specimens stored in air after 7 days, 28 days or 3 months moist curing, showed only small differences in total contraction with increase in MgO content of the cement where the MgO was substituted for CaO. An exception occurs in the case of the concrete specimens containing 9.9 per cent MgO which showed approximately 25 to 50 per cent greater total contraction than similar specimens containing 1.2 to 7 per cent MgO. The total contractions for this group of cements may have been influenced by the wide differences in the relative proportions of the C_3S and C_2S compounds. Where MgO was added, there was a trend toward reduced total contraction of concrete and mortar with increase in MgO (Fig. 10).

Resistance to Freezing and Thawing.—In general, the cements in which MgO was substituted for CaO as well as those in which it was added showed, in the freezing-and-thawing test, a tendency toward increased expansion and loss in weight as the MgO content of the cement increased (Fig. 10).

CONCLUSIONS

From the results presented in the preceding discussion the following conclusions are indicated:

1. *Strength:*

(*a*) The principal strength-giving constituents of portland cement are the tricalcium and dicalcium silicates. Tricalcium silicate not only contributed much of the strength at the early ages but continued to be effective in increasing strength at the later ages also. Dicalcium silicate contributed little to strength up to about 28 days but was largely responsible for the increase in strength beyond this period.

(*b*) Tricalcium aluminate contributed to strength to an important degree up to 28 days but its effect then diminished and became zero or negative at 1 and 2 years.

(*c*) The rôle of tetracalcium alumino ferrite is not so clearly indicated. It contributed to some extent to tensile strength after 1 day and to compressive strength of plastic mortars of 1:2.75 mix and of concrete at the 28-day and later periods.

2. *Contraction in Air:*

Tricalcium silicate and dicalcium silicate contributed to approximately the same degree to the total contraction, but to a much lesser degree than tricalcium aluminate and to a somewhat greater degree than tetracalcium alumino ferrite.

3. *Resistance to Sulfate Solutions:*

(*a*) Of the four major compounds, tricalcium aluminate appeared to be the only one which reduced the resistance of mortar and concrete to 2-per-cent solutions of magnesium and sodium sulfate. However, specimens exposed for a period of 3 years showed good resistance to the magnesium sulfate solution when the tricalcium aluminate content of the cement did not exceed 6 or 7 per cent and to the sodium sulfate solution when it did not exceed 6 to 10 per cent.

(*b*) In general, there was a noticeable decrease in the expansion of cylinders stored in the sulfate solutions with decrease in the tricalcium aluminate content of the cement. Where the tricalcium aluminate content was 7 per cent or less the expansions during the 2 years covered by the observations were not excessive. The expansion in water also tended to decrease with decrease in tricalcium aluminate.

4. *Resistance to Freezing and Thawing:*

On the basis of the expansions and losses in weight exhibited by concrete cylinders subjected to an accelerated freezing-and-thawing test, the higher the total content of tricalcium and dicalcium silicate of the cement the greater the resistance of the concrete. On the other hand, the greater the tricalcium aluminate and tetracalcium alumino ferrite content of the cement, the lower the resistance. The effect of tricalcium aluminate in lowering the resistance was indicated to be considerably greater than that of the tetracalcium alumino ferrite.

5. *Effect of MgO:*

(*a*) *Strength:*

Substitutions of MgO for CaO in the raw mix produced cements which showed at all ages a gradual decrease in strength with increase in MgO content. This apparent reduction in strength was largely due to the change in the relative proportions of tricalcium silicate and dicalcium silicate, and to some extent to the reduction in total silicates with increase in MgO content.

The addition of MgO in the raw mix, with the tricalcium silicate, tricalcium aluminate and tetracalcium alumino ferrite in the finished cement kept essentially constant, produced little effect on the strength of either mortars or concrete except for MgO contents of 7 per cent or more where decreased strength occurred at the 1 and 2-year periods. The reduction in total silicates resulting from the addition of the MgO was a contributing factor to the loss in strength which occurred.

(*b*) ***Expansion in Water:***

Concrete stored in water for 20 months showed increased expansion with increase in the MgO content of the cement, regardless of whether MgO was added to the raw mix or substituted for CaO.

(*c*) ***Expansion in Sulfate Solutions:***

Concrete stored in 2-per-cent solutions of magnesium and sodium sulfate up to 20 months, exhibited excessive expansion where the MgO content was greater than 5 or 6 per cent in the case of concrete stored in magnesium sulfate, and when greater than 7 per cent in the case of concrete stored in sodium sulfate.

(*d*) ***Contraction in Air:***

When MgO was substituted for CaO in the raw mix, only small differences in total contraction occurred except for the highest percentage. When MgO was added, there was a trend toward reduced contraction with increase in MgO. Changes in the proportions of the two silicates may have been a factor in these differences although the other data do not support this explanation.

(*e*) ***Resistance to Freezing and Thawing:***

Concrete subjected to alternate freezing and thawing showed a tendency toward increased expansion and greater loss in weight as the MgO content of the cement increased.

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TABLE I.—OXIDE ANALYSIS AND COMPUTED COMPOSITION OF CLINKERS.

The cements in this table are arranged in six groups according to their C_3S content, the cements in each group being arranged in descending order of C_3A content.

In general the SO_3 content of the clinker was close to 0 per cent and did not exceed one-tenth of 1 per cent. In grinding, gypsum was added to give an SO_3 content in the finished cement of 1.8 per cent.

Titanium is considered as TiO_2 since its compound in cement is not known.

Cement	Oxide Analysis of Clinker, per cent							Computed Composition of Clinker, per cent							
								Cement Compounds					Uncombined Oxides		
	SiO_2	Fe_2O_3	Al_2O_3	Total CaO	MgO	TiO_2	Free CaO	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	TiO_2	Free CaO
COMPUTED C_3S CONTENT ABOVE 60 PER CENT															
No. 104[a]	16.8	3.9	9.9	64.3	4.4	0.7	0	62	1	20	12	0	4.4	0.7	0
No. 106[a]	19.8	1.7	7.4	66.6	4.0	0.6	1.4	63	10	17	5	0	4.0	0.6	1.4
No. 102[a]	18.2	3.2	8.3	66.6	3.1	0.6	2.0	64	4	17	10	0	3.1	0.6	2.0
No. 38A[a]	21.1	2.2	6.6	66.4	3.3	0.5	0.3	61	14	14	7	0	3.3	0.5	0.3
No. 58[a]	20.6	2.4	6.8	66.4	3.3	0.5	0.7	62	13	14	7	0	3.3	0.5	0.7
No. 103[a]	18.3	5.5	7.5	64.7	3.4	0.6	0	66	3	11	17	0	3.4	0.6	0
No. 42	21.9	4.0	5.0	65.7	3.1	0.4	0.2	61	17	7	12	0	3.1	0.4	0.2
No. 108[a]	20.0	5.5	5.4	65.4	3.3	0.4	0	70	5	5	17	0	3.3	0.4	0
No. 62[a]	21.4	5.9	3.2	66.2	3.1	0.2	0.4	74	5	0	15	2	3.1	0.2	0.4
COMPUTED C_3S CONTENT 51 TO 60 PER CENT															
No. 100[a]	18.8	3.2	8.5	64.7	4.2	0.6	0.2	58	10	17	10	0	4.2	0.6	0.2
No. 105[a]	16.8	6.4	8.5	62.5	5.2	0.6	0	60	3	12	20	0	5.2	0.6	0
No. 40[a]	21.9	3.1	6.0	65.4	3.1	0.5	0	55	21	11	9	0	3.1	0.5	0
No. 101	18.9	5.6	7.4	64.2	3.4	0.6	0.7	56	12	10	17	0	3.4	0.6	0.7
No. 49	18.6	6.9	7.7	63.1	3.2	0.6	0.6	51	14	9	21	0	3.2	0.6	0.6
No. 9[a]	22.5	4.1	5.1	64.9	3.0	0.3	0	53	25	7	13	0	3.0	0.3	0
No. 60	21.6	4.0	5.1	66.0	3.0	0.4	2.3	55	20	7	12	0	3.0	0.4	2.3
No. 59	22.5	4.1	5.0	65.1	2.9	0.4	0.6	52	26	6	13	0	2.9	0.4	0.6
No. 60A	21.9	4.3	5.2	65.3	2.8	0.4	0.2	57	20	6	13	0	2.8	0.4	0.2
No. 10[a]	22.8	4.6	4.2	65.1	3.0	0.2	0.1	56	23	3	14	0	3.0	0.2	0.1
No. 44	22.5	4.8	4.1	65.3	3.0	0.3	0.2	59	20	3	15	0	3.0	0.3	0.2
No. 5B Dupl.	23.1	5.0	4.1	64.2	3.1	0.2	0.2	51	28	2	15	0	3.1	0.2	0.2
No. 11	23.5	5.9	3.1	64.2	3.0	0.2	0.1	51	29	0	15	2	3.0	0.2	0.1
No. 46	23.0	5.9	3.3	64.5	3.2	0.2	0.1	56	24	0	16	1	3.2	0.2	0.1
No. 61	23.0	5.7	3.4	64.5	3.1	0.3	0.1	56	24	0	16	1	3.1	0.3	0.1
COMPUTED C_3S CONTENT 41 TO 50 PER CENT															
No. 1 Dupl.	21.6	1.2	8.2	65.6	2.9	0.5	0.2	45	28	20	4	0	2.9	0.5	0.2
No. 1	21.9	1.2	7.7	66.1	2.8	0.5	1.4	43	27	18	4	0	2.8	0.5	1.4
No. 2B	21.5	2.2	7.5	65.2	3.0	0.5	0.3	47	26	16	7	0	3.0	0.5	0.3
No. 8 Dupl.	22.4	2.3	7.0	64.7	3.1	0.5	0.1	42	33	15	7	0	3.1	0.5	0.1
No. 2B Dupl.	22.5	2.2	6.9	65.0	2.9	0.5	0.2	43	32	15	7	0	2.9	0.5	0.2
No. 57	21.5	2.3	7.0	65.8	2.9	0.5	2.1	45	28	15	7	0	2.9	0.5	2.1
No. 47	20.8	3.1	7.8	64.8	3.0	0.6	0.2	48	24	15	9	0	3.0	0.6	0.2
No. 48	19.9	4.9	8.0	63.5	3.2	0.5	0.3	45	23	13	15	0	3.2	0.5	0.3
No. 12	21.8	3.9	7.1	64.0	2.8	0.4	0.1	41	32	12	12	0	2.8	0.4	0.1
No. 20	21.7	4.1	7.0	64.0	2.8	0.4	0	42	30	12	13	0	2.8	0.4	0
No. 34	23.4	3.1	6.3	65.5	1.4	0.4	0.2	41	36	11	9	0	1.4	0.4	0.2
No. 35	22.3	3.1	6.2	63.2	4.9	0.4	0.2	41	33	11	9	0	4.9	0.4	0.2
No. 3B	22.7	3.2	6.1	64.4	3.1	0.5	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 3B Dupl.	22.7	3.2	6.1	64.4	3.1	0.5	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 28	23.7	2.1	5.6	65.5	2.8	0.3	0.3	44	34	11	6	0	2.8	0.3	0.3
No. 31	22.9	3.1	6.1	66.3	1.2	0.4	0.3	49	29	11	9	0	1.2	0.4	0.3
No. 53[a]	24.6	1.3	4.7	66.1	3.0	0.3	0.3	47	35	10	4	0	3.0	0.3	0.3
No. 54	19.6	6.1	7.6	63.3	2.9	0.5	0.1	48	19	10	19	0	2.9	0.5	0.1
No. 16	24.4	2.1	4.8	65.6	2.9	0.3	0.9	42	38	9	6	0	2.9	0.3	0.9
No. 22	22.1	4.9	6.3	63.4	2.9	0.4	0	41	33	8	15	0	2.9	0.4	0
No. 24	23.6	4.0	5.2	64.1	2.8	0.3	0	41	37	7	12	0	2.8	0.3	0
No. 4	23.1	4.1	5.4	64.0	3.1	0.5	0	43	34	7	13	0	3.1	0.5	0
No. 18	24.9	3.0	3.9	65.1	2.8	0.3	0.4	43	39	5	9	0	2.8	0.3	0.4
No. 25	22.6	5.8	5.2	63.6	2.5	0.3	0	44	32	4	18	0	2.5	0.3	0
No. 50[a]	25.2	2.7	3.3	65.5	3.0	0.2	0.5	47	37	4	8	0	3.0	0.2	0.5
No. 15	21.0	6.7	5.9	63.1	2.8	0.4	0	48	24	4	20	0	2.8	0.4	0
No. 26	24.0	4.9	4.3	63.9	2.7	0.2	0	42	38	3	15	0	2.7	0.2	0
No. 27	23.0	6.6	4.5	62.8	2.9	0.2	0	41	35	1	20	0	2.9	0.2	0
No. 6	24.5	6.0	2.9	63.3	3.0	0.2	0	41	39	0	14	3	3.0	0.2	0
No. 7	24.5	7.1	2.1	63.2	3.0	0.1	0	42	39	0	10	6	3.0	0.1	0
No. 19	23.1	6.9	4.1	63.2	2.5	0.3	0	44	33	0	19	1	2.5	0.3	0
COMPUTED C_3S CONTENT 31 TO 40 PER CENT															
No. 30	21.3	4.4	7.6	63.4	2.8	0.5	0.1	39	32	13	13	0	2.8	0.5	0.1
No. 39A	24.0	3.0	6.0	63.7	3.0	0.4	0.3	31	46	11	9	0	3.0	0.4	0.3
No. 29	22.0	4.1	6.9	63.9	2.8	0.4	0	40	33	11	13	0	2.8	0.4	0
No. 32	23.3	2.9	5.8	62.9	4.7	0.3	0	36	40	10	9	0	4.7	0.3	0
No. 21	20.8	5.7	7.4	62.9	2.8	0.4	0	40	30	10	17	0	2.8	0.4	0
No. 14	22.9	4.8	5.9	63.3	2.8	0.4	0	37	38	7	15	0	2.8	0.4	0
No. 13	20.7	6.9	7.1	62.2	2.7	0.4	0	38	31	7	21	0	2.7	0.4	0
No. 23	21.5	6.7	6.3	62.3	2.8	0.4	0	38	33	5	20	0	2.8	0.4	0
No. 17	23.1	5.9	4.9	63.0	2.9	0.3	0	39	37	3	18	0	2.9	0.3	0
No. 51	25.5	4.1	3.2	63.7	3.3	0.3	0	38	45	2	13	0	3.3	0.3	0
No. 5B	24.2	5.2	3.9	63.3	3.1	0.2	0.1	39	40	2	16	0	3.1	0.2	0.1
COMPUTED C_3S CONTENT 21 TO 30 PER CENT															
No. 8[a]	23.8	2.2	6.9	63.2	3.3	0.6	0	27	48	15	7	0	3.3	0.	0
No. 41	24.7	3.9	5.0	62.9	3.0	0.4	0	29	49	7	12	0	3.0	0.4	0
No. 43	25.4	4.8	4.3	62.2	3.1	0.3	0	24	55	3	15	0	3.1	0.3	0
No. 45	25.5	5.9	3.2	62.1	3.1	0.2	0	28	52	0	15	2	3.1	0.2	0
COMPUTED C_3S CONTENT BELOW 21 PER CENT															
No. 37	24.5	2.1	7.2	62.6	3.0	0.5	0.2	16	58	16	6	0	3.0	0.5	0.2

[a] These cements were double burned.

TABLE II.—COMPRESSIVE STRENGTHS AND RATINGS OF 3 BY 6-IN. CONCRETE CYLINDERS.

Mix: 1:2.4:3.6 by weight.
Net water content 6.35 gal. per sack cement; water-cement ratio 0.847.
Aggregate: Elgin sand and gravel graded 0 to ¾ in.
Concrete placed in molds in 3 layers, each layer rodded 25 times with a ⅜-in. bullet-pointed steel rod.

Curing: 1 day in mold, then in moist room until 28 days old and then in water, air outdoors, or in sulfate solutions until tested at age indicated. The actual time the strength specimens were in water, air, or in the solutions was 28 days less than the age at test. All specimens tested wet. Air-stored specimens soaked in water for 48 hr. before test.

Ratings: A rating of 10 denotes that the specimen is in good condition, a rating of 9 that slight attack has occurred with the corners of the specimen beginning to crumble, while a rating of 8 indicates appreciable attack with about 15 per cent of the specimen disintegrated. Lower ratings denote greater degree of attack and a rating of zero indicates complete disintegration.

In general each strength value is the average of 3 tests and each rating value is the average of 9 to 15 specimens.

See Table I for composition of cements.

Cement	Compressive Strength at Age Indicated, lb. per sq. in.					Rating After Storage in Solutions for Periods Indicated											
						2 per cent $MgSO_4$ Solution						2 per cent Na_2SO_4 Solution					
	Moist Room, 28 days	Water, 1 year	Air Outdoors, 1 year[b]	$MgSO_4$ Solution, 1 year[b]	Na_2SO_4 Solution, 1 year[b]	6 months	1 year	1½ years	2 years	2½ years	3 years	6 months	1 year	1½ years	2 years	2½ years	3 years
COMPUTED C_3S CONTENT ABOVE 60 PER CENT																	
No. 104[a]	4360	5980	4580(77)	Disintegrated	Disintegrated	8.4	4.7	....	....	....	....	8.5	4.6	....	....	....	....
No. 106[a]	4740	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....
No. 102[a]	4820	5540	4560(82)	Disintegrated	Disintegrated	8.9	6.8	....	....	....	....	8.8	7.5	....	....	....	....
No. 38A[a]	4560	6070	5400(89)	Disintegrated	1620(27)	9.0	8.5	7.0	....	....	....	9.6	9.1	8.6	....	....	....
No. 58[a]	4540	5980	5060(85)	Disintegrated	5800(97)	9.7	8.7	6.0	2.0	....	....	9.7	9.2	8.9	8.2	....	....
No. 103[a]	4680	5080	4540(89)	3210(63)	5300(104)	10.0	9.3	....	....	....	....	10.0	9.8	....	....	....	....
No. 42	3730	5120	5150(100)	5230(102)	4850(95)	10.0	10.0	9.3	9.2	9.1	....	10.0	10.0	10.0	10.0	10.0	10.0
No. 108[a]	4090	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....
No. 62[a]	3500	4990	4610(92)	5310(106)	5300(106)	10.0	10.0	10.0	....	....	....	10.0	10.0	10.0	....	....	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT																	
No. 100[a]	4140	5190	4360(84)	Disintegrated	Disintegrated	9.0	6.8	....	....	....	....	9.1	8.0	....	....	....	....
No. 105[a]	3720	5420	4280(79)	Disintegrated	Disintegrated	9.7	8.6	....	....	....	....	9.3	8.8	....	....	....	....
No. 40[a]	4260	5130	4490(88)	4270(83)	4980(97)	10.0	9.2	8.4	8.0	....	....	10.0	9.6	9.1	8.6	....	....
No. 101	4390	5630	4690(83)	3070(54)	5020(89)	9.7	8.9	....	....	....	....	9.8	9.6	....	....	....	....
No. 49	4460	5750	4930(86)	1630(28)	5610(98)	9.8	9.3	8.4	5.2	....	....	10.0	10.0	9.7	9.2	....	....
No. 9[a]	3780	5060	4180(83)	4480(88)	5550(110)	10.0	9.4	9.3	9.0	8.0	6.3	10.0	10.0	10.0	10.0	10.0	10.0
No. 60	3730	5360	5230(98)	5680(106)	5720(107)	10.0	10.0	9.8	....	....	....	10.0	10.0	10.0	....	....	....
No. 59	3520	5250	4910(93)	5160(98)	5650(108)	10.0	10.0	9.9	9.1	....	....	10.0	10.0	9.9	9.9	....	....
No. 60A	4160	6040	5690(94)	5730(95)	6050(100)	10.0	10.0	9.3	....	....	....	10.0	10.0	10.0	....	....	....
No. 10[a]	3120	4800	4210(88)	4800(100)	5280(110)	10.0	9.0	9.0	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 44	2990	4610	4120(90)	4880(106)	4940(107)	10.0	10.0	10.0	9.4	....	....	10.0	10.0	10.0	10.0	....	....
No. 5B Dupl.	2780	4720	4490(95)	5190(110)	5430(115)	10.0	10.0	10.0	9.2	....	....	10.0	10.0	10.0	10.0	....	....
No. 11	3400	5420	4270(79)	4850(90)	5230(97)	10.0	9.0	9.0	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 46	3220	4620	4650(101)	4870(106)	5220(113)	10.0	9.6	9.2	9.1	9.0	....	10.0	10.0	10.0	10.0	10.0	....
No. 61	3220	5420	4600(85)	4740(87)	5170(95)	10.0	9.8	9.5	9.1	....	....	10.0	10.0	10.0	10.0	....	....

COMPUTED C_3S CONTENT 41 TO 50 PER CENT																	
No. 1 Dupl.	3690	5210	4420(85)	Disintegrated	Disintegrated	9.0	8.0	6.6	2.6	0.3	0	8.5	5.7	0.8	0	0	0
No. 1	3720	4660	4100(88)	Disintegrated	Disintegrated	9.2	5.1	0.9	0	0	0	8.4	5.5	0.7	0	0	0
No. 2B	4520	5460	4620(85)	Disintegrated	Disintegrated	10.0	7.0	2.4	0.8	0	0	8.6	7.6	5.0	2.0	1.2	0.5
No. 8 Dupl.	3690	5340	4900(92)	3720(70)	3510(66)	9.8	9.1	8.2	7.0	4.3	....	9.8	9.1	8.5	7.1	6.1	....
No. 2B Dupl.	4310	6100	4530(74)	Disintegrated	2990(49)	9.3	8.9	8.4	5.1	....	....	9.5	9.1	8.3	8.0	....	....
No. 57	4200	5840	5260(90)	Disintegrated	Disintegrated	9.1	8.0	5.0	....	....	....	9.1	8.3	6.8	....	....	....
No. 47	4280	5060	4220(83)	Disintegrated	Disintegrated	8.8	4.6	0.5	0	0	0	7.1	1.4	0.2	0	0	0
No. 48	4400	6150	5170(84)	Disintegrated	1930(31)	9.7	9.1	6.8	3.4	....	....	9.9	9.2	8.7	7.7	....	....
No. 12	4360	5690	4640(82)	3140(55)	6150(108)	10.0	8.8	8.0	6.9	3.7	0.7	10.0	10.0	9.7	9.3	9.1	8.8
No. 20	4430	5920	4930(83)	Disintegrated	5980(101)	9.9	8.7	7.5	6.3	3.3	2.1	10.0	9.7	9.3	9.2	8.8	8.3
No. 34	4500	5960	5530(93)	3650(61)	5380(90)	10.0	9.2	8.9	8.2	7.0	....	10.0	9.5	9.2	8.8	8.5	....
No. 35	4120	5310	4870(92)	1630(31)	5700(107)	10.0	9.3	8.9	7.2	5.4	....	9.9	9.7	9.3	8.9	8.7	....
No. 3B	4130	6040	4920(81)	2890(48)	1510(25)	9.9	8.8	6.4	3.9	2.0	1.0	9.9	8.1	7.1	6.2	6.2	6.0
No. 3B Dupl.	3770	5880	4480(76)	Disintegrated	3720(63)	9.5	8.7	4.3	1.4	0.2	0	9.9	9.4	7.7	6.4	5.9	5.2
No. 28	4130	5130	4870(95)	4940(96)	5280(103)	10.0	9.6	9.2	9.1	9.0	....	10.0	10.0	9.9	9.5	9.1	....
No. 31	4740	5800	4970(86)	5560(96)	3600(62)	10.0	9.7	9.0	8.5	7.6	....	10.0	9.8	9.2	9.0	8.6	....
No. 53[a]	4090	5850	5240(90)	4770(82)	5930(102)	10.0	9.5	9.1	9.0	....	....	10.0	10.0	10.0	10.0	....	....
No. 54	4170	5680	5010(88)	3060(54)	5630(99)	9.9	9.2	5.9	1.9	....	....	10.0	10.0	9.4	8.9	....	....
No. 16	3440	5150	4530(88)	4490(87)	5300(103)	10.0	9.0	8.9	8.5	8.0	6.2	10.0	10.0	10.0	10.0	9.8	9.8
No. 22	3520	5170	4840(94)	5700(110)	5890(114)	10.0	10.0	9.9	9.8	9.7	9.0	10.0	10.0	10.0	10.0	10.0	9.9
No. 24	3140	5030	4830(96)	5070(101)	5600(111)	10.0	10.0	9.6	9.4	9.2	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 4	3370	5310	4470(84)	5400(102)	5860(110)	10.0	10.0	10.0	10.0	9.2	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 18	3150	5490	4620(84)	5560(102)	4970(91)	10.0	9.9	9.8	9.7	9.1	9.1	10.0	10.0	10.0	10.0	10.0	10.0
No. 25	3740	5030	4870(97)	4850(96)	5250(105)	10.0	10.0	9.7	9.4	9.2	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 50[a]	2960	5000	4330(87)	4830(97)	5300(106)	10.0	10.0	10.0	9.2	....	....	10.0	10.0	10.0	10.0	....	....
No. 15	3440	4810	4420(92)	4880(102)	5520(115)	10.0	9.0	9.0	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 26	3290	4700	4670(99)	4900(104)	5750(122)	10.0	10.0	9.4	9.3	9.2	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 27	2940	4930	4620(94)	5040(102)	5160(104)	10.0	10.0	9.3	9.3	9.1	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 6	2170	4670	3800(81)	4730(101)	4950(106)	10.0	10.0	10.0	9.3	9.0	9.0	10.0	10.0	10.0	9.8	9.8	9.8
No. 7	2450	4340	3380(78)	4310(99)	4580(106)	10.0	10.0	9.1	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 19	2590	5070	4030(79)	4360(86)	5280(104)	10.0	9.0	9.0	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	9.9
COMPUTED C_3S CONTENT 31 TO 40 PER CENT																	
No. 30	3790	5510	4820(88)	4990(90)	1700(31)	10.0	9.3	8.8	8.3	7.4	....	10.0	9.4	8.2	6.9	4.7	....
No. 39A	3980	6170	5280(86)	1190(19)	5890(95)	9.0	8.6	7.1	....	....	....	10.0	9.3	9.2	....	....	....
No. 29	3550	5310	4390(83)	5610(106)	5320(100)	10.0	9.5	9.1	8.9	8.5	....	10.0	9.6	9.3	8.8	8.7	....
No. 32	4050	5670	4830(85)	4660(82)	5930(105)	10.0	9.9	9.1	8.5	7.5	....	10.0	10.0	9.7	9.5	9.3	....
No. 21	3870	5390	4790(89)	5460(101)	5340(99)	10.0	10.0	9.8	9.4	9.1	9.0	10.0	10.0	10.0	10.0	9.7	9.4
No. 14	3460	5200	4460(86)	4840(93)	5990(115)	10.0	9.0	9.0	8.6	7.7	5.5	10.0	10.0	9.9	9.8	9.7	9.5
No. 13	2840	5070	3790(75)	3510(69)	5280(104)	10.0	9.0	9.0	9.0	9.0	8.3	10.0	10.0	10.0	10.0	10.0	10.0
No. 23	3150	5330	4610(87)	5320(100)	5930(111)	10.0	10.0	9.7	9.7	9.3	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 17	3320	5050	4210(83)	4880(96)	5400(107)	10.0	9.0	9.0	9.0	9.0	9.0	10.0	10.0	10.0	10.0	10.0	10.0
No. 51	2910	5080	4560(90)	5040(99)	5460(108)	10.0	9.9	9.4	9.1	....	....	10.0	10.0	10.0	10.0	....	....
No. 5B	2680	5020	4250(85)	5290(105)	5700(114)	10.0	10.0	10.0	10.0	9.8	9.0	10.0	10.0	10.0	10.0	10.0	10.0
COMPUTED C_3S CONTENT 21 TO 30 PER CENT																	
No. 8[a]	3180	4620	3640(79)	Disintegrated	Disintegrated	9.6	6.4	0.8	0	0	0	9.5	8.3	6.3	2.1	0.3	0
No. 41	2890	5370	4780(89)	5120(95)	5650(106)	10.0	9.7	9.3	9.1	9.1	....	10.0	10.0	10.0	10.0	10.0	....
No. 43	1980	4340	4200(97)	4510(104)	5020(116)	10.0	9.6	9.1	9.0	9.0	....	10.0	10.0	10.0	10.0	10.0	....
No. 45	1850	4340	3820(88)	4290(99)	4710(108)	10.0	9.3	9.2	9.1	9.0	....	10.0	10.0	9.9	9.8	9.8	....
COMPUTED C_3S CONTENT BELOW 21 PER CENT																	
No. 37	3230	4830	4570(95)	Disintegrated	Disintegrated	9.2	7.3	4.0	1.0	0	0	8.8	5.3	1.2	0	0	0

[a] These cements were double burned.

[b] Values in parentheses are the strengths of specimens stored in the sulfate solutions expressed as percentages of the strength of similar specimens stored in water and tested at the same age.

TABLE III.—COMPRESSIVE STRENGTH OF 2-IN. PLASTIC MORTAR CUBES OF 1:2.75 MIX BY WEIGHT.

Sand: Run-of-mine Ottawa silica sand of the following average grading:
Passing No. 14 sieve, 100 per cent; No. 28 sieve, 95 per cent; No. 48 sieve, 28 per cent; No. 100 sieve, 5 per cent (Fineness Modulus, 1.72).
Water content 6 gal. per sack cement; water-cement ratio 0.80.
Mortar placed in molds in 2 layers, each layer compacted by puddling with fingers.
Cubes tested at ages up to 28 days were stored in moist room; all others were stored in moist room for 28 days, then in water or in sulfate solutions until tested at age indicated. The actual time in water or in the solutions was 28 days less than the age at test. All specimens tested wet.
In general each strength value is the average of 3 tests.
See Table I for composition of cements and Table IV for ratings in sulfate solutions.

Cement	Compressive Strength at Age Indicated, lb. per sq. in.												
	Stored in Moist Room				Stored in Moist Room 28 Days, Remainder as Indicated								
					In Water			In 2 per cent $MgSO_4$ Solution			In 2 per cent Na_2SO_4 Solution		
	1 day	3 days	7 days	28 days	3 months	1 year	2 years	3 months^b	1 year^b	2 years^b	3 months^b	1 year^b	2 years^b
COMPUTED C_3S CONTENT ABOVE 60 PER CENT													
No. 104^a	270	1360	2430	4050	4200	3590	....	3820(91)	Disintegrated	Disintegrated	3380(80)	Disintegrated	Disintegrated
No. 106^a	870	2650	4100	6060	6170	....	....	5570(90)	....	....	5930(96)	....	....
No. 102^a	770	2130	3410	4270	4480	4050	....	3250(73)	Disintegrated	Disintegrated	4190(94)	Disintegrated	Disintegrated
No. 38A^a	580	1970	3310	4890	5390	5500	....	5040(94)	Disintegrated	Disintegrated	5300(98)	Disintegrated	Disintegrated
No. 58^a	630	1860	3280	4820	5110	4980	5290	5020(98)	Disintegrated	Disintegrated	5630(110)	Disintegrated	Disintegrated
No. 103^a	580	1820	3170	4570	5390	4670	....	5220(97)	Disintegrated	Disintegrated	5380(100)	Disintegrated	Disintegrated
No. 42	530	1710	2750	4380	5640	6220	5710	5560(99)	5630(90)	5090(89)	5790(103)	6520(105)	5950(104)
No. 108^a	570	1800	2670	4850	5920	....	....	5740(97)	....	....	5990(101)	....	....
No. 62^a	450	1900	2920	3940	5000	5510	....	5470(109)	5460(99)	....	5150(103)	5520(100)	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT													
No. 100^a	460	1550	2530	4090	3880	3520	....	2820(73)	Disintegrated	Disintegrated	2970(77)	Disintegrated	Disintegrated
No. 105^a	290	1060	1650	3350	4210	4260	....	2760(66)	Disintegrated	Disintegrated	3300(78)	Disintegrated	Disintegrated
No. 40^a	610	1650	2890	4770	5360	6180	6270	5490(102)	810(13)	....	5420(101)	1070(17)	Disintegrated
No. 101	610	1580	2780	4560	4820	4660	....	4950(103)	Disintegrated	Disintegrated	4410(92)	Disintegrated	Disintegrated
No. 49	400	1210	2270	3870	4490	4460	4450	4490(100)	2490(56)	Disintegrated	4790(107)	3700(83)	1030(23)
No. 9^a	490	1460	2310	4760	6440	6720	6580	6750(105)	3720(55)	2680(41)	6580(102)	6190(92)	4920(75)
No. 60	670	1540	2410	4120	5460	5470	....	5690(101)	4370(80)	....	5500(98)	5330(97)	....
No. 59	420	1300	2040	3950	4960	5220	5730	5130(103)	4980(95)	4520(79)	4920(99)	5320(102)	5720(100)
No. 60A	520	1730	2530	4200	5660	5780	....	5900(104)	5280(91)	....	5880(104)	5770(100)	....
No. 10^a	420	1520	2290	4440	6550	6590	6790	6330(97)	6270(95)	5450(80)	5880(90)	7400(112)	7100(105)
No. 44	360	1360	2100	3120	4290	5170	5360	4550(106)	5110(99)	5320(99)	4850(113)	4960(96)	5850(109)
No. 5B Dupl	360	1090	1660	2880	4260	4950	5250	4580(107)	5130(103)	5240(100)	4700(110)	5430(109)	6090(116)
No. 11	370	1500	2330	4020	5510	5980	6130	5920(107)	5810(97)	5950(97)	5940(108)	6770(113)	6580(107)
No. 46	410	1450	2190	3720	4720	5200	5700	4820(102)	5750(110)	5860(103)	5030(107)	5750(111)	6160(108)
No. 61	340	1280	1990	3410	4540	5280	5770	5030(111)	5180(98)	5070(88)	4740(104)	5410(102)	5750(100)

COMPUTED C_3S CONTENT 41 TO 50 PER CENT

No 1 Dupl.	570	2000	2960	4310	4970	4720	4800	4410(89)	Disintegrated	Disintegrated	4520(91)	Disintegrated	Disintegrated
No. 1	760	2000	2890	4560	5500	5210	5550	4190(76)	Disintegrated	Disintegrated	4660(85)	Disintegrated	Disintegrated
No. 2B	560	1680	2730	4890	5300	6130	5640	4290(81)	Disintegrated	Disintegrated	5320(100)	Disintegrated	Disintegrated
No. 8 Dupl.	420	1340	2440	4450	5960	5000	6400	5360(90)	Disintegrated	Disintegrated	5910(99)	Disintegrated	Disintegrated
No. 2B Dupl.	480	1430	2410	4320	5870	5380	5490	5450(93)	Disintegrated	Disintegrated	4960(84)	Disintegrated	Disintegrated
No. 57	580	1610	2510	4010	5750	5530	....	3700(64)	Disintegrated	Disintegrated	4380(76)	Disintegrated	Disintegrated
No. 47	610	1660	2720	4270	5210	5110	5200	4380(84)	Disintegrated	Disintegrated	3520(68)	Disintegrated	Disintegrated
No. 48	370	1310	2340	4170	5080	5060	4850	5000(98)	Disintegrated	Disintegrated	5260(103)	Disintegrated	Disintegrated
No. 12	500	1340	2230	4610	5920	6250	6060	5660(96)	Disintegrated	Disintegrated	5630(95)	Disintegrated	Disintegrated
No. 20	330	1220	2310	4720	5890	5870	5710	5990(102)	Disintegrated	Disintegrated	6080(103)	Disintegrated	Disintegrated
No. 34	450	1210	2020	4580	5840	6350	6120	5060(87)	Disintegrated	Disintegrated	6300(108)	Disintegrated	Disintegrated
No. 35	440	1220	1770	4020	5180	5550	5960	5350(103)	Disintegrated	Disintegrated	5480(106)	Disintegrated	Disintegrated
No. 3B	460	1260	2010	4460	6440	6930	6710	6020(94)	Disintegrated	Disintegrated	5850(91)	Disintegrated	Disintegrated
No. 3B Dupl.	460	1190	1900	4090	5760	6280	6120	5550(96)	Disintegrated	Disintegrated	5430(94)	Disintegrated	Disintegrated
No. 28	510	1380	2300	4370	5780	5780	6380	5400(93)	1300(22)	Disintegrated	5900(102)	3220(56)	Disintegrated
No. 31	580	1480	2400	4470	6080	5930	6280	5350(88)	Disintegrated	Disintegrated	6280(103)	Disintegrated	Disintegrated
No. 53[a]	450	1150	2240	3910	4920	5570	5790	5070(103)	4050(73)	Disintegrated	4810(98)	5140(92)	2900(50)
No. 54	410	1240	2120	3750	4540	4260	4490	4790(105)	Disintegrated	Disintegrated	4580(101)	Disintegrated	Disintegrated
No. 16	540	1390	2260	4460	5730	6190	6560	5820(102)	Disintegrated	Disintegrated	6010(105)	5240(85)	Disintegrated
No. 22	340	1280	2320	4730	5650	6930	6680	5920(105)	5170(75)	3750(56)	5960(105)	6310(90)	4740(71)
No. 24	400	1360	2020	4480	6450	6850	6790	6320(98)	5650(82)	5310(78)	6100(95)	6470(94)	6970(103)
No. 4	490	1270	2170	4690	6390	6820	7070	6440(101)	5280(77)	4560(64)	6760(106)	6240(92)	5720(81)
No. 18	390	1330	2110	4250	5930	6820	7120	6170(104)	5970(88)	5570(78)	6350(107)	6880(101)	6900(97)
No. 25	340	1270	2110	4400	5650	6370	5980	5910(105)	6110(96)	5750(96)	6070(107)	6920(109)	6890(115)
No. 50[a]	300	990	1590	2860	4960	6300	6400	4900(99)	5750(91)	6080(95)	5020(101)	5850(93)	6400(100)
No. 15	300	1260	2210	4610	5660	6870	6920	6260(111)	5940(86)	5490(79)	6260(111)	7020(102)	6790(98)
No. 26	310	1100	1810	4200	6330	6970	7450	5850(92)	6340(91)	6370(86)	5900(93)	7050(101)	7340(99)
No. 27	240	980	1640	3470	5370	5980	6040	4960(92)	6440(108)	5340(88)	5560(104)	6250(104)	6340(105)
No. 6	240	810	1210	2820	5470	6060	6570	5490(100)	5950(98)	5830(89)	5270(96)	6590(109)	6650(101)
No. 7	200	790	1220	2760	4800	5610	5940	5170(108)	5930(106)	5830(98)	5300(110)	6410(114)	6570(111)
No. 19	200	890	1490	3190	5170	6210	6730	5440(105)	5650(91)	6490(96)	5500(106)	6510(105)	6200(92)

COMPUTED C_3S CONTENT 31 TO 40 PER CENT

No. 30	300	1100	2060	3890	5130	5520	5230	4710(92)	Disintegrated	Disintegrated	5040(98)	Disintegrated	Disintegrated
No. 39A	380	880	1430	3710	5660	6060	....	4440(78)	Disintegrated	Disintegrated	4870(86)	Disintegrated	Disintegrated
No. 29	260	890	1670	3490	4710	5290	5160	4260(90)	Disintegrated	Disintegrated	4680(99)	Disintegrated	Disintegrated
No. 32	430	1170	1900	4480	5650	5970	6190	5130(91)	Disintegrated	Disintegrated	5270(93)	Disintegrated	Disintegrated
No. 21	330	1330	2320	4300	5660	6140	5660	5790(102)	3640(59)	1580(28)	5840(103)	5020(82)	3970(70)
No. 14	360	1210	2180	4200	5470	6400	6700	5800(106)	3140(49)	2080(31)	5980(109)	6430(100)	4650(69)
No. 13	250	820	1620	3680	5410	6060	6360	5490(101)	4180(69)	3620(57)	5490(101)	5780(95)	5490(86)
No. 23	270	910	1680	3520	5470	6250	6440	5180(100)	5190(83)	4980(77)	5310(97)	6200(99)	6180(96)
No. 17	260	1100	1910	3730	5770	6680	6880	6040(105)	5920(89)	5710(83)	6160(107)	7350(110)	7320(106)
No. 51	210	850	1330	2770	5150	6090	6060	5180(100)	6410(105)	6380(105)	5320(103)	6300(103)	6250(103)
No. 5B	310	900	1260	3300	5560	6410	6860	5890(106)	5780(90)	5340(78)	5860(105)	6670(104)	6750(98)

COMPUTED C_3S CONTENT 21 TO 30 PER CENT

No. 8[a]	270	900	1370	3280	4870	4990	5180	3630(75)	Disintegrated	Disintegrated	4550(93)	Disintegrated	Disintegrated
No. 41	280	780	1330	3440	5760	6100	5710	5770(100)	5500(90)	4440(78)	5730(100)	6880(113)	6650(116)
No. 43	180	510	710	2500	4750	5800	6060	5710(120)	6200(107)	5390(89)	5680(120)	6080(105)	6470(107)
No. 45	150	560	920	2540	5630	6510	6300	5770(102)	6360(105)	6480(103)	5730(102)	6630(102)	6290(100)

COMPUTED C_3S CONTENT BELOW 21 PER CENT

No. 37	310	660	1190	3470	5340	5370	5550	4930(92)	Disintegrated	Disintegrated	5200(97)	Disintegrated	Disintegrated

[a] These cements were double burned.

[b] Values in parentheses are the strengths of specimens stored in the sulfate solutions expressed as percentages of the strength of similar specimens stored in water and tested at the same age.

TABLE IV.—RATINGS OF 2-IN. PLASTIC MORTAR CUBES OF 1:2.75 MIX STORED IN SULFATE SOLUTIONS.

Curing: 1 day in mold, then in moist room until 28 days old and then in the sulfate solutions. The ratings of the specimens are based on the actual time in the solutions. The age of the specimens is 28 days more than the time in the solutions.

Ratings: A rating of 10 denotes that the specimen is in good condition, a rating of 9 that slight attack has occurred with the corners of the specimen beginning to crumble, while a rating of 8 indicates appreciable attack with about 15 per cent of the specimen disintegrated. Lower ratings denote greater degree of attack and a rating of zero indicates complete disintegration.

In general each value is the average of 9 to 15 specimens.

For further details see Table III.

For cement compositions see Table I.

Cement	Ratings After Storage in Sulfate Solutions for Periods Indicated											
	In 2 per cent $MgSO_4$ Solution						In 2 per cent Na_2SO_4 Solution					
	6 months	1 year[b]	1½ years	2 years[b]	2½ years	3 years[b]	6 months	1 year[b]	1½ years	2 years[b]	2½ years	3 years[b]
COMPUTED C_3S CONTENT ABOVE 60 PER CENT												
No. 104[a]	7.1	0(7.1)	0	0(7.1)	0	0(7.1)	6.3	0(6.3)	0	0(6.3)	0	0(6.3)
No. 106[a]	...	....	...	....	...	....	...	....	...	....	...	....
No. 102[a]	6.3	1.5(7.8)	0	0(7.8)	0	0(7.8)	7.0	1.2(8.2)	0	0(8.2)	0	0(8.2)
No. 38A[a]	7.7	5.3(13.0)	...	....	...	....	8.4	8.0(16.4)	...	....	...	....
No. 58[a]	8.7	3.4(12.1)	0	0(12.1)	0	0(12.1)	8.5	3.4(11.9)	0.4	0(12.3)	0	0(12.3)
No. 103[a]	8.1	2.0(10.1)	0	0(10.1)	0	0(10.1)	8.1	4.5(12.6)	...	....	...	....
No. 42	10	9.8(19.8)	9.0	9.0(37.8)	8.8	....	10	10 (20.0)	9.4	9.1(38.5)	9.0	....
No. 108[a]	...	....	...	....	...	....	...	....	...	....	...	....
No. 62[a]	10	10 (20.0)	10	....	...	....	10	10 (20.0)	10	....	...	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT												
No. 100[a]	6.8	1.5(8.3)	0	0(8.3)	0	0(8.3)	7.1	0.7(7.8)	0	0(7.8)	0	0(7.8)
No. 105[a]	6.9	0(6.9)	0	0(6.9)	0	0(6.9)	6.0	0(6.0)	0	0(6.0)	0	0(6.0)
No. 40[a]	9.2	7.4(16.6)	1.7	0(18.3)	0	0(18.3)	9.1	5.6(14.7)	1.1	0(15.8)	0	0(15.8)
No. 101	7.9	1.6(9.5)	0	0(9.5)	0	0(9.5)	7.8	0.9(8.7)	0	0(8.7)	0	0(8.7)
No. 49	9.0	7.8(16.8)	1.6	0(18.4)	0	0(18.4)	9.9	9.2(19.1)	8.7	8.2(36.0)	...	....
No. 9[a]	10	9.8(19.8)	9.0	9.0(37.8)	6.9	1.1(45.8)	10	10 (20.0)	9.7	9.1(38.8)	8.0	8.0(54.8)
No. 60	10	9.6(19.6)	9.2	....	...	....	10	10 (20.0)	10	....	...	....
No. 59	9.6	9.0(18.6)	9.0	9.0(36.6)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 60A	10	9.9(19.9)	9.2	....	...	....	9.8	9.7(19.5)	9.6	....	...	....
No. 10[a]	10	9.8(19.8)	9.0	9.0(37.8)	9.0	9.0(55.8)	10	10 (20.0)	10	9.6(39.6)	8.9	8.9(57.4)
No. 44	10	10 (20.0)	9.8	9.1(38.9)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B Dupl	10	10 (20.0)	9.9	....	...	....	10	10 (20.0)	10	....	...	....
No. 11	10	9.8(19.8)	9.0	9.0(37.8)	9.0	9.0(55.8)	10	10 (20.0)	9.9	9.6(39.5)	9.4	9.1(58.0)
No. 46	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 61	9.6	9.0(18.6)	9.0	9.0(36.6)	...	....	10	10 (20.0)	10	10 (40.0)	...	....

COMPUTED C_3S CONTENT 41 TO 50 PER CENT												
No. 1 Dupl.	7.6	2.7(10.3)	0	0(10.3)	0	0(10.3)	8.1	2.0(10.1)	0	0(10.1)	0	0(10.1)
No. 1	8.4	3.1(11.5)	0	0(11.5)	0	0(11.5)	9.0	3.0(12.0)	0	0(12.0)	0	0(12.0)
No. 2B	9.1	1.9(11.0)	0	0(11.0)	0	0(11.0)	9.0	3.0(12.0)	0	0(12.0)	0	0(12.0)
No. 8 Dupl.	7.5	2.7(10.2)	0	0(10.2)	0	0(10.2)	8.3	7.3(15.6)	1.8	0(17.4)	0	0(17.4)
No. 2B Dupl.	8.7	4.6(13.3)	0	0(13.3)	0	0(13.3)	8.9	4.2(13.1)	0	0(13.1)	0	0(13.1)
No. 57	5.9	0.1(6.0)	0	0(6.0)	0	0(6.0)	7.7	1.3(9.0)	0	0(9.0)	0	0(9.0)
No. 47	7.2	2.0(9.2)	0	0(9.2)	0	0(9.2)	5.1	0(5.1)	0	0(5.1)	0	0(5.1)
No. 48	8.7	3.7(12.4)	0	0(12.4)	0	0(12.4)	8.1	2.2(10.3)	0	0(10.3)	0	0(10.3)
No. 12	7.6	3.3(10.9)	0	0(10.9)	0	0(10.9)	9.7	3.5(13.2)	0	0(13.2)	0	0(13.2)
No. 20	7.9	3.2(11.1)	0	0(11.1)	0	0(11.1)	10	2.7(12.7)	0	0(12.7)	0	0(12.7)
No. 34	7.7	1.3(9.0)	0	0(9.0)	0	0(9.0)	8.2	0(8.2)	0	0(8.2)	0	0(8.2)
No. 35	8.2	2.3(10.5)	0	0(10.5)	0	0(10.5)	8.3	0(8.3)	0	0(8.3)	0	0(8.3)
No. 3B	9.0	3.6(12.6)	0	0(12.6)	0	0(12.6)	8.5	0(8.5)	0	0(8.5)	0	0(8.5)
No. 3B Dupl.	8.8	3.8(12.6)	0	0(12.6)	0	0(12.6)	8.0	1.8(9.8)	0	0(9.8)	0	0(9.8)
No. 28	9.3	6.2(15.5)	2.7	0(18.2)	0	0(18.2)	10	8.1(18.1)	3.2	0(21.3)	0	0(21.3)
No. 31	8.5	2.2(10.7)	0	0(10.7)	0	0(10.7)	9.3	3.7(13.0)	0	0(13.0)	0	0(13.0)
No. 53[a]	10	8.2(18.2)	7.3	3.5(29.0)	...	....	10	9.3(19.3)	9.0	8.2(36.5)	...	....
No. 54	8.7	5.3(14.0)	0.2	0(14.2)	0	0(14.2)	8.5	3.0(11.5)	0.2	0(11.7)	0	0(11.7)
No. 16	9.0	7.7(16.7)	1.4	0(18.1)	0	0(18.1)	10	9.6(19.6)	8.3	7.7(35.6)	6.2	....
No. 22	9.9	9.5(19.4)	9.0	9.0(37.4)	8.0	....	10	9.8(19.8)	9.1	8.9(37.8)	8.7	....
No. 24	9.9	9.3(19.2)	9.0	9.0(37.2)	9.0	....	10	10 (20.0)	9.8	9.7(39.5)	9.3	....
No. 4	10	10 (20.0)	9.8	9.0(38.8)	9.0	9.0(56.8)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 18	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	10 (20.0)	9.3	9.1(38.4)	8.8	....
No. 25	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	9.8	9.8(39.6)	9.8	....
No. 50[a]	10	10 (20.0)	9.8	9.2(39.0)	...	....	10	10 (20.0)	9.8	9.7(39.5)	...	....
No. 15	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	10 (20.0)	9.9	9.4(39.3)	9.2	....
No. 26	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	9.8	9.8(39.6)	9.8	....
No. 27	10	9.5(19.5)	9.0	9.0(37.5)	9.0	....	10	9.8(19.8)	9.0	9.0(37.8)	9.0	....
No. 6	10	10 (20.0)	9.2	9.0(38.2)	9.0	9.0(56.2)	10	10 (20.0)	10	9.9(39.9)	9.9	9.9(59.7)
No. 7	10	10 (20.0)	10	9.0(39.0)	9.0	9.0(57.0)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 19	10	9.6(19.6)	9.0	9.0(37.6)	9.0	9.0(55.6)	10	10 (20.0)	9.8	9.0(38.8)	9.0	9.0(56.8)
COMPUTED C_3S CONTENT 31 TO 40 PER CENT												
No. 30	8.1	3.3(11.4)	0	0(11.4)	0	0(11.4)	8.9	3.3(12.2)	0	0(12.2)	0	0(12.2)
No. 39A	6.7	2.8(9.5)	0	0(9.5)	0	0(9.5)	8.0	4.0(12.0)	0	0(12.0)	0	0(12.0)
No. 29	8.6	3.0(11.6)	0	0(11.6)	0	0(11.6)	9.3	4.4(13.7)	0	0(13.7)	0	0(13.7)
No. 32	8.1	2.7(10.8)	0	0(10.8)	0	0(10.8)	9.4	4.0(13.4)	0	0(13.4)	0	0(13.4)
No. 21	10	9.4(19.4)	8.0	6.6(34.0)	4.5	....	10	9.6(19.6)	8.9	8.7(37.2)	8.6	....
No. 14	10	10 (20.0)	9.0	9.0(38.0)	7.1	3.1(48.2)	10	9.9(19.9)	9.3	9.0(38.2)	6.8	6.1(51.1)
No. 13	10	9.8(19.8)	9.0	9.0(37.8)	8.4	7.8(54.0)	10	10 (20.0)	9.3	9.0(38.3)	8.9	8.9(56.1)
No. 23	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	9.7	9.5(39.2)	9.3	....
No. 17	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	10 (20.0)	9.7	9.1(38.8)	9.0	....
No. 51	10	10 (20.0)	9.4	9.1(38.5)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B	10	10 (20.0)	9.8	9.0(38.8)	9.0	9.0(56.8)	10	10 (20.0)	10	10 (40.0)	10	9.9(59.9)
COMPUTED C_3S CONTENT 21 TO 30 PER CENT												
No. 8[a]	7.8	0(7.8)	0	0(7.8)	0	0(7.8)	9.0	0(9.0)	0	0(9.0)	0	0(9.0)
No. 41	10	9.8(19.8)	9.0	9.0(37.8)	8.1	....	10	10 (20.0)	9.1	9.0(38.1)	9.0	....
No. 43	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 45	10	9.7(19.7)	9.0	9.0(37.7)	9.0	....	10	9.7(19.7)	9.0	9.0(37.7)	9.0	....
COMPUTED C_3S CONTENT BELOW 21 PER CENT												
No. 37	8.7	3.5(12.2)	0	0(12.2)	0	0(12.2)	8.6	5.3(13.9)	0.8	0(14.7)	0	0(14.7)

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[a] These cements were double burned.

[b] Values in parentheses are "durability factors" and are the sums of all the individual ratings up to and including the period indicated.

TABLE V.—COMPRESSIVE STRENGTH OF 2-IN. PLASTIC MORTAR CUBES OF 1:4.25 MIX BY WEIGHT.

Water content 9 gal. per sack cement; water-cement ratio 1.20.
In general each strength value is the average of 3 tests.
For other details, see Tables III and VI.

Cement	Compressive Strength at Age Indicated, lb. per sq. in.												
	Stored in Moist Room				Stored in Moist Room 28 Days, Remainder as Indicated								
					In Water			In 2 per cent $MgSO_4$ Solution			In 2 per cent Na_2SO_4 Solution		
	1 day	3 days	7 days	28 days	3 months	1 year	2 years	3 months[b]	1 year[b]	2 years[b]	3 months[b]	1 year[b]	2 years[b]
COMPUTED C_3S CONTENT ABOVE 60 PER CENT													
No. 104[a]	70	420	800	1360	1430	1300	....	Disintegrated	Disintegrated	Disintegrated	Disintegrated	Disintegrated	Disintegrated
No. 106[a]	250	970	1620	2200	2410	....	....	450(19)	....	....	1870(78)	....	....
No. 102[a]	230	790	1230	1820	1840	1490	....	1010(55)	Disintegrated	Disintegrated	940(51)	Disintegrated	Disintegrated
No. 38A[a]	170	690	1430	1960	2150	1970	....	1100(51)	Disintegrated	Disintegrated	1460(68)	Disintegrated	Disintegrated
No. 58[a]	210	610	1190	1850	2080	2050	2180	1320(63)	Disintegrated	Disintegrated	1550(75)	Disintegrated	Disintegrated
No. 103[a]	170	550	1070	1730	1770	1510	....	1360(77)	Disintegrated	Disintegrated	1260(71)	Disintegrated	Disintegrated
No. 42	150	600	980	1610	2090	2250	2330	2280(109)	2710(120)	2550(109)	2160(103)	2740(122)	2740(118)
No. 108[a]	150	560	860	1340	2000	....	....	2110(105)	....	....	2000(100)	....	....
No. 62[a]	170	670	1040	1490	1890	2070	....	2230(118)	2530(122)	....	2140(113)	2380(115)	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT													
No. 100[a]	150	560	1050	1640	1490	1460	....	880(59)	Disintegrated	Disintegrated	930(62)	Disintegrated	Disintegrated
No. 105[a]	80	310	490	1070	1460	1560	....	760(52)	Disintegrated	Disintegrated	880(60)	Disintegrated	Disintegrated
No. 40[a]	190	530	920	1820	2270	2220	2610	1880(83)	1300(59)	Disintegrated	1640(72)	720(32)	Disintegrated
No. 101	170	470	920	1670	1640	1450	....	980(60)	Disintegrated	Disintegrated	1090(66)	Disintegrated	Disintegrated
No. 49	130	440	770	1360	1520	1350	1300	1190(78)	Disintegrated	Disintegrated	1510(99)	250(16)	Disintegrated
No. 9[a]	140	480	880	1850	2730	2510	2410	2950(108)	1730(69)	1470(61)	2690(99)	2860(114)	2060(86)
No. 60	230	570	820	1460	1960	1900	....	2230(114)	1760(93)	....	2070(106)	1980(104)	....
No. 59	140	460	640	1140	1700	2080	2260	2090(123)	2180(105)	2090(92)	1900(112)	2260(109)	2360(104)
No. 60A	190	600	870	1550	1960	2160	....	2260(115)	2380(110)	....	2410(123)	2240(104)	....
No. 10[a]	140	470	720	1450	2470	2850	2920	2700(109)	2880(101)	2430(83)	2630(106)	3080(108)	3190(109)
No. 44	100	480	690	1060	1730	2010	2180	1910(110)	2260(112)	2520(115)	1810(105)	2450(122)	2650(122)
No. 5B Dupl.	130	410	580	1040	1530	1790	2120	1750(114)	2140(120)	2650(125)	1670(109)	2220(124)	2520(119)
No. 11	120	500	750	1410	2110	2740	2690	2240(106)	2850(104)	2990(111)	2360(112)	2990(109)	3130(116)
No. 46	170	550	830	1320	1930	2050	2210	2100(109)	2410(118)	2400(109)	2070(107)	2280(111)	2490(113)
No. 61	110	430	620	1140	1680	2130	2290	1900(113)	2350(110)	2710(118)	1920(114)	2310(108)	2540(111)
COMPUTED C_3S CONTENT 41 TO 50 PER CENT													
No. 1 Dupl.	220	770	1240	1800	2290	2170	2270	1660(72)	Disintegrated	Disintegrated	1830(80)	Disintegrated	Disintegrated
No. 1	270	800	1200	1830	2420	2370	2210	1350(56)	Disintegrated	Disintegrated	2030(84)	Disintegrated	Disintegrated
No. 2B	160	680	1150	2250	2680	2480	2460	1410(53)	Disintegrated	Disintegrated	2140(80)	Disintegrated	Disintegrated
No. 8 Dupl.	120	480	1010	1950	2840	2500	2850	1900(67)	Disintegrated	Disintegrated	2120(75)	970(39)	Disintegrated
No. 2B Dupl.	150	570	940	1840	2380	2330	2290	860(36)	Disintegrated	Disintegrated	1840(77)	Disintegrated	Disintegrated
No. 57	200	540	850	1710	1970	2370	....	Disintegrated	Disintegrated	Disintegrated	1300(66)	Disintegrated	Disintegrated
No. 47	180	540	860	1660	2010	1990	2070	Disintegrated	Disintegrated	Disintegrated	Disintegrated	Disintegrated	Disintegrated
No. 48	110	420	750	1490	1780	1530	1650	920(52)	Disintegrated	Disintegrated	1380(78)	Disintegrated	Disintegrated
No. 12	140	460	860	1760	2510	2440	2530	1790(71)	Disintegrated	Disinetgrated	1950(78)	Disintegrated	Disintegrated
No. 20	100	420	860	1860	2460	2560	2770	1980(81)	Disintegrated	Disintegrated	2290(93)	Disintegrated	Disintegrated
No. 34	140	410	710	1780	2630	2750	2570	1710(65)	Disintegrated	Disintegrated	2510(95)	Disintegrated	Disintegrated
No. 35	130	410	770	1810	2240	2410	2310	1320(59)	Disintegrated	Disintegrated	1980(88)	Disintegrated	Disintegrated
No. 3B	130	500	880	1990	2920	3190	2900	2240(77)	Disintegrated	Disintegrated	2530(87)	Disintegrated	Disintegrated
No. 3B Dupl.	130	470	760	1690	2470	2830	2890	2140(87)	Disintegrated	Disintegrated	2530(102)	Disintegrated	Disintegrated
No. 28	160	450	820	1610	2320	2660	2600	2030(88)	Disintegrated	Disintegrated	2410(104)	900(34)	....
No. 31	160	430	900	1850	2490	2440	2300	1920(77)	Disintegrated	Disintegrated	2560(103)	Disintegrated	Disintegrated
No. 53[a]	130	420	680	1550	2170	2550	2680	2210(102)	1460(57)	Disintegrated	2250(104)	280(11)	Disintegrated
No. 54	140	410	740	1300	1550	1730	1570	1170(76)	Disintegrated	Disintegrated	1510(97)	Disintegrated	Disintegrated
No. 16	170	480	820	1800	2530	2850	2840	2910(115)	Disintegrated	Disintegrated	2570(102)	2230(78)	Disintegrated
No. 22	120	390	820	1720	2360	2720	2740	2400(102)	2230(82)	1880(69)	2310(98)	2700(99)	2610(95)
No. 24	140	400	640	1440	2690	2840	2560	2660(99)	2810(99)	2840(111)	2740(102)	3110(109)	3120(122)
No. 4	130	460	730	1630	2650	3080	3040	2630(99)	1900(62)	1770(58)	2600(98)	2960(96)	2440(80)
No. 18	150	370	640	1550	2520	3010	3140	2690(107)	3130(104)	2770(88)	2680(106)	3360(112)	3400(108)
No. 25	120	410	660	1440	2130	2470	2700	2240(105)	2820(114)	3120(116)	2250(106)	2850(115)	3160(117)
No. 50[a]	90	400	550	1110	1970	2640	2610	1960(100)	2750(104)	2750(105)	2030(103)	2500(95)	3130(120)
No. 15	100	410	730	1520	2220	2620	2670	2260(102)	3010(115)	2780(104)	2370(107)	2930(112)	3160(118)
No. 26	100	350	500	1370	2560	2840	2890	2460(96)	3060(108)	3250(112)	2490(97)	3060(108)	3500(121)
No. 27	80	320	530	1070	2080	2360	2510	2140(103)	2690(114)	2810(112)	2110(101)	2610(111)	2960(118)
No. 6	80	270	400	890	2160	2810	2800	2270(105)	2950(105)	2990(107)	2250(104)	3170(113)	3060(109)
No. 7	60	300	440	1020	1880	2720	2600	1950(104)	2980(110)	2650(102)	1940(103)	3020(111)	2890(111)
No. 19	70	330	510	1160	2130	2990	3120	2180(102)	3310(111)	3310(106)	2270(107)	3360(112)	3340(107)
COMPUTED C_3S CONTENT 31 TO 40 PER CENT													
No. 30	90	320	580	1350	2090	2070	2200	1480(71)	Disintegrated	Disintegrated	1950(93)	Disintegrated	Disintegrated
No. 39A	110	300	480	1400	1990	2420	....	780(39)	Disintegrated	Disintegrated	1820(91)	Disintegrated	Disintegrated
No. 29	80	310	530	1210	2070	2350	2290	1780(86)	Disintegrated	Disintegrated	2010(97)	990(42)	....
No. 32	130	360	620	1580	2630	2850	2730	1880(71)	Disintegrated	Disintegrated	2600(99)	Disintegrated	Disintegrated
No. 21	120	400	830	1720	2270	2310	2230	2260(100)	1470(64)	1260(56)	2330(103)	2350(102)	2190(98)
No. 14	110	380	700	1500	2460	2680	2770	2420(98)	1500(56)	1220(44)	2750(112)	2530(94)	2030(73)
No. 13	80	280	600	1270	2080	2680	2590	2280(110)	2060(77)	1950(75)	2200(106)	2810(105)	2530(98)
No. 23	100	310	520	1070	2290	2510	2610	2250(98)	2840(113)	2800(107)	2350(103)	2930(117)	3280(126)
No. 17	70	380	620	1390	2220	2870	2830	2330(105)	2870(100)	3350(118)	2330(105)	2950(103)	3430(121)
No. 51	60	280	430	1000	1920	2400	2560	1990(104)	2860(119)	2610(102)	1800(94)	2640(110)	2800(109)
No. 5B	90	310	450	1080	2220	2870	2770	2430(109)	3210(112)	2570(93)	2380(107)	3150(110)	3490(126)
COMPUTED C_3S CONTENT 21 TO 30 PER CENT													
No. 8[a]	80	340	550	1310	2510	2750	2610	1880(75)	Disintegrated	Disintegrated	2130(85)	Disintegrated	Disintegrated
No. 41	80	240	390	1170	2370	2830	2780	2410(102)	2510(89)	2240(81)	2590(109)	2800(99)	2670(96)
No. 43	80	210	260	870	1970	2260	2470	2090(106)	2470(109)	2810(114)	2210(112)	2510(111)	2930(118)
No. 45	50	160	260	770	2170	2700	2710	2260(104)	2850(105)	2980(110)	2280(105)	2560(95)	3030(112)
COMPUTED C_3S CONTENT BELOW 21 PER CENT													
No. 37	100	250	380	1130	2240	2170	2100	1930(86)	Disintegrated	Disintegrated	2320(103)	Disintegrated	Disintegrated

[a] These cements were double burned.

[b] Values in parentheses are the strengths of specimens stored in the sulfate solutions expressed as percentages of the strength of similar specimens stored in water and tested at the same age.

TABLE VI.—RATINGS OF 2-IN. PLASTIC MORTAR CUBES OF 1:4.25 MIX STORED IN SULFATE SOLUTIONS.

For computed compositions and other details see notes accompanying Tables I, III and V.

Cement	Ratings After Storage in Sulfate Solutions for Periods Indicated											
	In 2 per cent $MgSO_4$ Solution						In 2 per cent Na_2SO_4 Solution					
	6 mo.	1 year[b]	1½ yr.	2 years[b]	2½ yr.	3 years[b]	6 mo.	1 year[b]	1½ yr.	2 years[b]	2½ yr.	3 years[b]
COMPUTED C_3S CONTENT ABOVE 60 PER CENT												
No. 104[a]	2.5	0(2.5)	0	0(2.5)	0	0(2.5)	0.2	0(0.2)	0	0(0.2)	0	0(0.2)
No. 106[a]	...	....	...	....	...	....	...	....	...	....	...	....
No. 102[a]	2.7	0(2.7)	0	0(2.7)	0	0(2.7)	2.4	0(2.4)	0	0(2.4)	0	0(2.4)
No. 38A[a]	4.0	0(4.0)	0	0(4.0)	0	0(4.0)	1.2	0(1.2)	0	0(1.2)	0	0(1.2)
No. 58[a]	4.9	0(4.9)	0	0(4.9)	0	0(4.9)	1.5	0(1.5)	0	0(1.5)	0	0(1.5)
No. 103[a]	3.5	0(3.5)	0	0(3.5)	0	0(3.5)	4.7	1.2(5.9)	...	....	...	....
No. 42	10	9.2(19.2)	9.0	9.0(37.2)	9.0	....	10	10 (20.0)	9.1	9.0(38.1)	9.0	....
No. 108[a]	...	....	...	....	...	....	...	....	...	....	...	....
No. 62[a]	9.9	9.9(19.8)	9.3	....	...	....	10	10 (20.0)	10	....	...	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT												
No. 100[a]	3.0	0(3.0)	0	0(3.0)	0	0(3.0)	0.2	0(0.2)	0	0(0.2)	0	0(0.2)
No. 105[a]	3.4	0(3.4)	0	0(3.4)	0	0(3.4)	2.7	0(2.7)	0	0(2.7)	0	0(2.7)
No. 40[a]	9.2	7.8(17.0)	1.2	0(18.2)	0	0(18.2)	7.8	1.9(9.7)	0	0(9.7)	0	0(9.7)
No. 101	3.6	0(3.6)	0	0(3.6)	0	0(3.6)	2.4	0(2.4)	0	0(2.4)	0	0(2.4)
No. 49	7.5	2.2(9.7)	0	0(9.7)	0	0(9.7)	8.9	7.9(16.8)	1.5	0(18.3)	0	0(18.3)
No. 9[a]	10	9.9(19.9)	9.0	9.0(37.9)	9.0	9.0(55.9)	10	10 (20.0)	9.4	9.0(38.4)	8.9	8.4(55.7)
No. 60	9.9	9.1(19.0)	9.0	....	...	....	9.9	9.3(19.2)	9.3	....	...	....
No. 59	9.5	9.1(18.6)	9.0	9.0(36.6)	...	....	9.3	9.0(18.3)	9.0	9.0(36.3)	...	....
No. 60A	9.9	9.5(19.4)	9.0	....	...	....	10	9.7(19.7)	9.7	....	...	....
No. 10[a]	10	9.8(19.8)	9.0	9.0(37.8)	9.0	9.0(55.8)	10	10 (20.0)	9.8	9.2(39.0)	9.0	9.0(57.0)
No. 44	10	10 (20.0)	9.4	9.0(38.4)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B Dupl.	10	10 (20.0)	10	9.5(39.5)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 11	10	9.8(19.8)	9.0	9.0(37.8)	9.0	9.0(55.8)	10	10 (20.0)	10	9.7(39.7)	9.0	9.0(57.7)
No. 46	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	10	10 (40.0)	9.0	....
No. 61	9.3	9.2(18.5)	9.0	9.0(36.5)	...	....	9.3	9.0(18.3)	9.0	9.0(36.3)	...	....
COMPUTED C_3S CONTENT 41 TO 50 PER CENT												
No. 1 Dupl.	6.0	0.6(6.6)	0	0(6.6)	0	0(6.6)	6.0	1.1(7.1)	0	0(7.1)	0	0(7.1)
No. 1	6.0	0(6.0)	0	0(6.0)	0	0(6.0)	4.0	0(4.0)	0	0(4.0)	0	0(4.0)
No. 2B	6.2	0(6.2)	0	0(6.2)	0	0(6.2)	4.0	0(4.0)	0	0(4.0)	0	0(4.0)
No. 8 Dupl.	5.0	0(5.0)	0	0(5.0)	0	0(5.0)	3.5	0(3.5)	0	0(3.5)	0	0(3.5)
No. 2B Dup.	7.4	1.0(8.4)	0	0(8.4)	0	0(8.4)	8.1	0.4(8.5)	0	0(8.5)	0	0(8.5)
No. 57	3.8	0(3.8)	0	0(3.8)	0	0(3.8)	3.7	0(3.7)	0	0(3.7)	0	0(3.7)
No. 47	4.5	0(4.5)	0	0(4.5)	0	0(4.5)	4.8	0.5(5.3)	0	0(5.3)	0	0(5.3)
No. 48	3.8	0(3.8)	0	0(3.8)	0	0(3.8)	3.2	0(3.2)	0	0(3.2)	0	0(3.2)
No. 12	6.2	0.6(6.8)	0	0(6.8)	0	0(6.8)	4.8	0.1(4.9)	0	0(4.9)	0	0(4.9)
No. 20	7.5	0.6(8.1)	0	0(8.1)	0	0(8.1)	8.8	0.8(9.6)	0	0(9.6)	0	0(9.6)
No. 34	5.7	0.5(6.2)	0	0(6.2)	0	0(6.2)	5.6	0.5(6.1)	0	0(6.1)	0	0(6.1)
No. 35	5.3	0(5.3)	0	0(5.3)	0	0(5.3)	4.7	0.5(5.2)	0	0(5.2)	0	0(5.2)
No. 3B	5.0	0(5.0)	0	0(5.0)	0	0(5.0)	4.0	0(4.0)	0	0(4.0)	0	0(4.0)
No. 3B Dupl.	4.8	0(4.8)	0	0(4.8)	0	0(4.8)	5.0	0(5.0)	0	0(5.0)	0	0 5.0)
No. 28	8.2	3.8(12.0)	0	0(12.0)	0	0(12.0)	9.9	4.2(14.1)	1.8	0(15.9)	0	0(15.9)
No. 31	5.6	0(5.6)	0	0(5.6)	0	0(5.6)	5.8	0.6(6.4)	0	0(6.4)	0	0((6.4)
No. 53[a]	9.3	5.0(14.3)	0	0(14.3)	0	0(14.3)	9.5	8.3(17.8)	5.1	1.1(24.0)	...	....
No. 54	6.0	0.6(6.6)	0	0(6.6)	0	0(6.6)	5.0	0(5.0)	0	0(5.0)	0	0(5.0)
No. 16	9.6	7.5(17.1)	1.2	0(18.3)	0	0(18.3)	10	8.6(18.6)	1.8	0(20.4)	0	0(20.4)
No. 22	10	9.6(19.6)	9.0	9.0(37.6)	9.0	9.0(55.6)	10	9.8(19.8)	9.6	9.2(38.6)	9.1	9.0(56.7)
No. 24	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	9.8(19.8)	9.7	9.3(38.8)	9.0	....
No. 4	10	10 (20.0)	10	10 (40.0)	9.0	9.0(58.0)	10	10 (20.0)	10	9.0(39.0)	9.0	9.0(57.0)
No. 18	10	9.6(19.6)	9.0	9.0(37.6)	9.0	9.0(55.6)	10	9.8(19.8)	9.5	9.1(38.4)	9.0	8.7(56.1)
No. 25	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	9.9(19.9)	9 7	9.2(38.8)	9.0	....
No. 50[a]	9.0	9.0(18.0)	9.0	9.0(36.0)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 15	10	9.6(19.6)	9.0	9.0(37.6)	9.0	9.0(55.6)	10	9.8(19.8)	9.3	9.0(38.1)	9.0	9.0(56.1)
No. 26	10	9.4(19.4)	9.0	9.0(37.4)	8.9	....	10	9.9(19.9)	9.7	9.7(39.3)	9.7	....
No. 27	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	9.0	9.0(38.0)	9.0	....
No. 6	10	10 (20.0)	9.4	9.3(38.7)	9.1	9.0(56.8)	10	10 (20.0)	9.7	9.1(38.8)	9.0	9.0(56.8)
No. 7	10	10 (20.0)	9.8	9.3(39.1)	9.1	9.0(57.2)	10	10 (20.0)	10	9.3(39.3)	9.1	9.0(57.4)
No. 19	10	9.6(19.6)	9.0	9.0(37.6)	9.0	9.0(55.6)	10	9.8(19.8)	9.4	9.4(38.6)	9.3	9.0(56.9)
COMPUTED C_3S CONTENT 31 TO 40 PER CENT												
No. 30	6.7	1.6(8.3)	0	0(8.3)	0	0(8.3)	9.4	3.0(12.4)	0	0(12.4)	0	0(12.4)
No. 39A	4.1	0.4(4.5)	0	0(4.5)	0	0(4.5)	7.8	0.6(8.4)	0	0(8.4)	0	0(8.4)
No. 29	8.8	4.4(13.2)	0	0(13.2)	0	0(13.2)	10	8.5(18.5)	2.5	0(21.0)	0	0(21.0)
No. 32	5.9	0(5.9)	0	0(5.9)	0	0(5.9)	8.8	2.1(10.9)	0	0(10.9)	0	0(10.9)
No. 21	10	9.6(19.6)	9.0	8.0(36.6)	1.4	0(38.0)	10	9.8(19.8)	9.5	9.0(38.3)	9.0	....
No. 14	10	9.6(19.6)	9.0	8.0(36.6)	6.6	6.2(49.4)	10	9.8(19.8)	9.1	9.0(37.9)	8.3	6.6(52.8)
No. 13	10	9.8(19.8)	9.0	9.0(37.8)	9.0	9.0(55.8)	10	10 (20.0)	9.7	9.1(38.8)	9.0	8.1(55.9)
No. 23	10	9.6(19.6)	9.0	9.0(37.6)	9.0	....	10	9.8(19.8)	9.7	9.3(38.8)	9.0	....
No. 17	10	9.4(19.4)	9.0	9.0(37.4)	9.0	9.0(55.4)	10	9.8(19.8)	9.7	9.4(38.9)	9.2	9.0(57.1)
No. 51	10	9.0(19.0)	9.0	9.0(37.0)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B	10	10 (20.0)	10	10 (40.0)	9.4	9.0(58.4)	10	10 (20.0)	10	9.0(39.0)	9.0	9.0(57.0)
COMPUTED C_3S CONTENT 21 TO 30 PER CENT												
No. 8[a]	6.0	0(6.0)	0	0(6.0)	0	0(6.0)	5.0	2.8(7.8)	0	0(7.8)	0	0(7.8)
No. 41	10	9.2(19.2)	9.0	9.0(37.2)	8.8	....	10	10 (20.0)	9.3	9.2(38.5)	9.0	....
No. 43	10	9.4(19.4)	9.0	9.0(37.4)	9.0	....	10	10 (20.0)	10	10 (40.0)	9.0	....
No. 45	10	9.0(19.0)	9.0	9.0(37.0)	9.0	....	10	9.9(19.9)	9.9	9.8(39.6)	9.4	....
COMPUTED C_3S CONTENT BELOW 21 PER CENT												
No. 37	7.2	0.7(7.9)	0	0(7.9)	0	0(7.9)	8.3	1.7(10.0)	0	0(10.0)	0	....

[a] These cements were double burned.

[b] Values in parentheses are "durability factors" and are the sums of all the individual ratings up to and including the period indicated.

TABLE VII.—TENSILE STRENGTH OF 1:3 STANDARD OTTAWA SAND BRIQUETS.

Briquets made and tested in accordance with A.S.T.M. Standard Methods of Sampling and Testing Portland Cement (C 77 - 32).

Briquets tested at ages up to 28 days were stored in moist closet; all others were stored in moist closet for 28 days, then in water or in sulfate solutions until tested at age indicated. The actual time in water or in the solutions was 28 days less than the age at test. All specimens tested wet.

In general each strength value is the average of 3 tests.

See Table I for composition of cements and Table VIII for ratings in sulfate solutions.

Cement	Tensile Strength at Age Indicated, lb. per sq. in.												
	Stored in Moist Closet				Stored in Moist Closet 28 Days, Remainder as Indicated								
					In Water			In 2 per cent $MgSO_4$ Solution			In 2 per cent Na_2SO_4 Solution		
	1 day	3 days	7 days	28 days	3 months	1 year	2 years	3 months[b]	1 year[b]	2 years[b]	3 months[b]	1 year[b]	2 years[b]
COMPUTED C_3S CONTENT ABOVE 60 PER CENT													
No. 104[a]	135	255	400	480	410	250	....	390(95)	Disintegrated	Disintegrated	520(127)	Disintegrated	Disintegrated
No. 106[a]	235	345	455	475	450	....	....	315(70)	....	....	505(112)	....	....
No. 102[a]	215	330	435	485	420	330	....	305(73)	Disintegrated	Disintegrated	435(104)	340(103)	....
No. 38A[a]	190	335	420	500	475	455	....	445(94)	Disintegrated	Disintegrated	565(119)	415(91)	....
No. 58[a]	205	355	430	505	425	420	355	475(112)	30(7)	Disintegrated	500(118)	445(106)	230(65)
No. 103[a]	170	330	400	425	410	295	....	285(70)	Disintegrated	Disintegrated	490(119)	150(51)	....
No. 42	170	325	385	450	460	485	460	460(100)	230(47)	70(15)	450(98)	505(104)	455(99)
No. 108[a]	165	330	405	480	480	....	....	490(102)	....	....	545(113)	....	....
No. 62[a]	160	325	405	470	460	450	....	470(102)	360(80)	....	520(113)	520(116)	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT													
No. 100[a]	185	350	455	495	445	350	....	340(76)	Disintegrated	Disintegrated	500(112)	205(59)	....
No. 105[a]	130	225	335	425	445	275	....	315(71)	Disintegrated	Disintegrated	385(87)	Disintegrated	Disintegrated
No. 40[a]	180	300	385	425	505	430	435	410(81)	45(10)	Disintegrated	460(91)	430(100)	360(83)
No. 101	195	350	420	475	445	345	....	280(63)	Disintegrated	Disintegrated	460(103)	320(93)	....
No. 49	170	315	365	460	435	345	330	425(98)	135(39)	....	550(126)	385(112)	265(80)
No. 9[a]	140	310	345	445	495	475	450	500(101)	90(19)	55(12)	560(113)	480(101)	420(93)
No. 60	185	270	310	410	350	335	....	320(91)	80(24)	....	505(144)	410(122)	....
No. 59	140	305	345	405	475	435	420	475(100)	175(40)	110(26)	540(114)	465(107)	400(95)
No. 60A	135	275	375	470	470	430	....	460(98)	135(31)	....	530(113)	440(102)	....
No. 10[a]	145	285	330	450	550	505	495	565(103)	295(58)	150(30)	580(105)	590(117)	540(109)
No. 44	115	270	335	420	470	450	480	425(90)	285(63)	155(32)	535(114)	515(114)	455(95)
No. 5B Dupl.	195	320	405	440	475	465	435	480(101)	330(71)	260(60)	485(102)	495(106)	495(114)
No. 11	120	245	290	470	450	460	500	520(116)	430(93)	355(71)	510(113)	570(124)	575(115)
No. 46	135	290	380	475	465	455	450	470(101)	415(91)	260(58)	520(112)	505(111)	515(114)
No. 61	125	295	340	395	445	425	390	485(109)	390(92)	350(90)	510(115)	495(117)	480(123)

COMPUTED C_3S CONTENT 41 TO 50 PER CENT													
No. 1 Dupl.	190	345	430	495	520	450	425	510(98)	Disintegrated	Disintegrated	525(101)	495(110)	185(44)
No. 1	170	320	410	475	505	435	410	465(92)	Disintegrated	Disintegrated	545(108)	395(91)	165(40)
No. 2B	165	285	405	515	510	465	430	520(102)	Disintegrated	Disintegrated	560(110)	335(72)	Disintegrated
No. 8 Dupl.	175	310	355	485	490	490	455	450(92)	225(46)	Disintegrated	505(103)	430(88)	Disintegrated
No. 2B Dupl.	190	310	375	480	500	485	430	425(85)	30(6)	Disintegrated	530(106)	500(103)	430(100)
No. 57	170	270	350	440	405	355	....	290(72)	Disintegrated	Disintegrated	510(126)	255(72)	....
No. 47	185	295	360	420	445	370	370	380(85)	Disintegrated	Disintegrated	485(109)	Disintegrated	Disintegrated
No. 48	180	305	370	460	480	380	360	485(101)	Disintegrated	Disintegrated	535(111)	430(113)	Disintegrated
No. 12	155	275	345	480	460	475	400	525(114)	Disintegrated	Disintegrated	525(114)	540(114)	Disintegrated
No. 20	140	320	385	460	500	470	475	535(107)	Disintegrated	Disintegrated	525(105)	555(118)	150(32)
No. 34	205	335	380	460	475	540	490	495(104)	165(31)	13(3)	530(112)	555(103)	570(116)
No. 35	160	260	330	435	435	405	360	435(100)	15(4)	Disintegrated	480(110)	260(64)	Disintegrated
No. 3B	150	275	355	455	525	390	455	530(101)	Disintegrated	Disintegrated	500(95)	540(139)	195(43)
No. 3B Dupl.	140	260	340	395	510	475	450	545(107)	Disintegrated	Disintegrated	585(115)	450(95)	160(36)
No. 28	205	335	395	465	530	505	490	490(92)	190(38)	70(14)	520(98)	555(110)	545(111)
No. 31	195	330	360	440	520	485	470	475(91)	105(22)	7(1)	565(109)	545(112)	570(121)
No. 53[a]	150	265	325	450	500	400	455	500(100)	195(49)	30(7)	540(108)	480(120)	415(91)
No. 54	185	340	380	455	435	385	365	430(99)	45(12)	Disintegrated	480(110)	405(105)	180(49)
No. 16	160	270	340	445	460	375	440	485(105)	60(16)	20(5)	515(112)	490(131)	480(109)
No. 22[a]	150	320	365	485	475	470	460	540(114)	175(37)	85(18)	500(105)	525(112)	510(111)
No. 24	140	300	335	445	495	525	500	540(109)	235(45)	110(22)	545(110)	565(108)	540(108)
No. 4	105	215	305	405	475	485	495	450(95)	165(34)	100(20)	495(104)	450(93)	315(64)
No. 18	140	275	335	450	475	505	515	485(102)	285(57)	190(37)	515(108)	530(105)	455(88)
No. 25	120	285	335	445	510	480	445	535(105)	270(56)	200(45)	525(103)	565(118)	525(118)
No. 50[a]	125	245	320	475	505	480	460	555(110)	365(76)	240(52)	555(110)	530(110)	490(107)
No. 15	125	315	350	470	495	455	470	530(107)	215(47)	175(37)	580(117)	565(124)	480(102)
No. 26	125	300	360	460	505	490	485	545(108)	315(64)	200(41)	535(106)	535(109)	515(106)
No. 27	145	300	380	425	500	480	475	485(97)	445(93)	305(64)	555(111)	490(102)	515(109)
No. 6	85	200	245	365	530	475	450	555(105)	425(89)	245(54)	585(111)	565(119)	515(114)
No. 7	75	190	270	345	485	455	440	505(104)	495(109)	315(72)	565(117)	515(113)	540(123)
No. 19	80	235	335	425	500	545	500	515(103)	495(91)	405(81)	540(108)	550(101)	575(115)
COMPUTED C_2S CONTENT 31 TO 40 PER CENT													
No. 30	135	305	370	450	465	485	445	460(99)	70(14)	Disintegrated	495(106)	525(108)	530(119)
No. 39A	130	240	290	440	505	530	....	505(100)	Disintegrated	Disintegrated	490(97)	505(95)	....
No. 29	135	300	350	445	490	480	475	495(101)	Disintegrated	Disintegrated	495(101)	555(116)	540(114)
No. 32	180	295	330	430	505	480	435	480(95)	95(20)	30(7)	505(100)	500(104)	465(107)
No. 21	160	310	370	435	485	485	470	515(106)	135(28)	65(14)	565(117)	490(101)	500(106)
No. 14	150	315	345	445	545	475	465	525(96)	90(19)	65(14)	570(105)	555(117)	490(105)
No. 13	115	245	345	480	530	470	435	570(108)	145(31)	105(24)	565(107)	540(115)	530(122)
No. 23	125	275	310	445	490	465	465	515(105)	290(62)	175(38)	485(99)	550(118)	485(104)
No. 17	110	295	350	445	490	470	475	575(117)	300(64)	205(43)	565(115)	550(117)	545(115)
No. 51	120	245	320	450	505	445	485	575(114)	420(94)	290(60)	510(101)	480(108)	535(110)
No. 5B	80	210	225	370	475	445	465	420(88)	270(61)	160(34)	565(119)	515(116)	500(108)
COMPUTED C_3S CONTENT 21 TO 30 PER CENT													
No. 8[a]	105	215	260	430	525	460	415	435(83)	Disintegrated	Disintegrated	455(87)	345(75)	Disintegrated
No. 41	120	240	320	425	500	465	465	545(109)	240(52)	75(16)	485(97)	525(113)	435(94)
No. 43	95	185	215	410	535	485	435	480(90)	365(75)	150(35)	510(95)	505(104)	430(99)
No. 45	65	180	245	415	505	485	460	555(110)	460(95)	265(58)	515(102)	505(104)	485(105)
COMPUTED C_3S CONTENT BELOW 21 PER CENT													
No. 37	120	205	275	410	475	480	445	465(98)	Disintegrated	Disintegrated	485(102)	Disintegrated	Disintegrated

[a] These cements were double burned.

[b] Values in parentheses are the strengths of specimens stored in the sulfate solutions expressed as percentages of the strength of similar specimens stored in water and tested at the same age.

TABLE VIII.—RATINGS OF 1:3 STANDARD SAND BRIQUETS STORED IN SULFATE SOLUTIONS.

For details of tests see notes accompanying Table IV.
For composition of cements see Table I, and for tensile strengths of briquets see Table VII.

Cement	Ratings After Storage in Sulfate Solutions for Periods Indicated											
	In 2 per cent $MgSO_4$ Solution						In 2 per cent Na_2SO_4 Solution					
	6 months	1 year^b	1½ years	2 years^b	2½ years	3 years^b	6 months	1 year^b	1½ years	2 years^b	2½ years	3 years^b
COMPUTED C_3S CONTENT ABOVE 60 PER CENT												
No. 104^a	6.8	0.2(7.0)	...	....	...	....	7.8	2.4(10.2)	...	....	...	....
No. 106^a	...	....	...	....	...	....	...	....	...	....	...	....
No. 102^a	7.4	1.4(8.8)	...	....	...	....	9.2	6.8(16.0)	...	....	...	....
No. 38A^a	8.3	3.6(11.9)	...	....	...	....	9.9	9.4(19.3)	...	....	...	....
No. 58^a	9.3	7.4(16.7)	1.5	0(18.2)	0	0(18.2)	10	9.5(19.5)	8.0	6.7(34.2)	...	....
No. 103^a	8.3	1.2(9.5)	...	....	...	....	10	8.1(18.1)	...	....	...	....
No. 42	10	9.4(19.4)	9.0	9.0(37.4)	8.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 108^a	...	....	...	....	...	....	...	....	...	....	...	....
No. 62^a	9.7	8.7(18.4)	8.3	....	...	....	10	10 (20.0)	10	....	...	....
COMPUTED C_3S CONTENT 51 TO 60 PER CENT												
No. 100^a	8.1	2.6(10.7)	...	....	...	....	9.7	7.8(17.5)	...	....	...	....
No. 105^a	5.6	0(5.6)	0	0(5.6)	0	0(5.6)	7.3	1.1(8.4)	0	0(8.4)	0	0(8.4)
No. 40^a	10	7.0(17.0)	1.6	0(18.6)	0	0(18.6)	10	9.7(19.7)	9.7	9.2(38.6)	...	....
No. 101	8.3	1.2(9.5)	0	0(9.5)	0	0(9.5)	9.9	9.1(19.0)	...	....	...	....
No. 49	9.9	9.2(19.1)	3.8	0.7(23.6)	...	....	10	10 (20.0)	9.8	8.5(38.3)	...	....
No. 9^a	10	10 (20.0)	9.0	9.0(38.0)	0.7	0(38.7)	10	10 (20.0)	10	10 (40.0)	10	9.7(59.7)
No. 60	9.6	8.6(18.2)	6.9	....	...	....	10	10 (20.0)	10	....	...	....
No. 59	9.6	9.0(18.6)	8.6	8.2(35.4)	...	....	10	10 (20.0)	10	9.9(39.9)	...	....
No. 60A	9.5	8.5(18.0)	7.9	....	...	....	10	10 (20.0)	10	....	...	....
No. 10^a	10	10 (20.0)	9.0	9.0(38.0)	8.5	7.1(53.6)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 44	10	9.0(19.0)	9.0	8.9(36.9)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B Dupl.	10	9.8(19.8)	9.1	....	...	....	10	10 (20.0)	10	....	...	....
No. 11	10	10 (20.0)	9.0	9.0(38.0)	8.8	8.0(54.8)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 46	10	9.5(19.5)	9.0	9.0(37.5)	9.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 61	9.9	9.7(19.6)	8.6	8.1(36.3)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
COMPUTED C_3S CONTENT 41 TO 50 PER CENT												
No. 1 Dupl.	9.2	3.8(13.0)	0.3	0(13.3)	0	0(13.3)	10	9.3(19.3)	8.6	6.9(34.8)	4.1	....
No. 1	9.5	3.9(13.4)	1.3	0.7(15.4)	0	0(15.4)	9.8	8.0(17.8)	6.1	5.4(29.3)	5.1	2.9(37.3)
No. 2B	9.6	2.5(12.1)	0	0(12.1)	0	0(12.1)	9.8	7.2(17.0)	6.0	4.0(27.0)	1.2	0(28.2)
No. 8 Dupl.	9.5	5.9(15.4)	1.8	0(17.2)	0	0(17.2)	9.9	9.1(19.0)	7.5	4.7(31.2)	3.2	....
No. 2B Dupl.	10	6.3(16.3)	2.1	0(18.4)	0	0(18.4)	10	10 (20.0)	10	9.8(39.8)	...	....
No. 57	6.6	0.2(6.8)	0	0(6.8)	0	0(6.8)	9.6	8.3(17.9)	5.2	....	...	....
No. 47	5.9	1.1(7.0)	0	0(7.0)	0	0(7.0)	9.3	8.2(17.5)	3.2	0(20.7)	0	0(20.7)
No. 48	9.8	7.0(16.8)	0.6	0(17.4)	0	0(17.4)	9.9	9.5(19.4)	6.6	4.5(30.5)	...	....
No. 12	9.8	6.1(15.9)	1.1	0(17.0)	0	0(17.0)	10	9.6(19.6)	7.8	5.7(33.1)	2.7	1.0(36.8)
No. 20	9.6	5.1(14.7)	0.4	0(15.1)	0	0(15.1)	10	9.6(19.6)	8.8	8.3(36.7)	3.3	3.3(43.3)
No. 34	10	9.0(19.0)	5.2	3.0(27.2)	0.8	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 35	9.6	3.8(13.4)	0	0(13.4)	0	0(13.4)	10	9.2(19.2)	6.2	0.2(25.6)	0	0(25.6)
No. 3B	9.8	7.0(16.8)	0	0(16.8)	0	0(16.8)	10	9.7(19.7)	8.2	4.8(32.7)	3.3	3.0(39.0)
No. 3B Dupl.	9.9	6.4(16.3)	3.0	0(19.3)	0	0(19.3)	10	9.4(19.4)	6.2	3.7(29.3)	2.0	1.5(32.8)
No. 28	10	9.0(19.0)	9.0	9.0(37.0)	7.3	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 31	10	8.9(18.9)	5.5	2.3(26.7)	0	0(26.7)	10	10 (20.0)	10	10 (40.0)	10	....
No. 53^a	9.8	9.1(18.9)	4.7	1.5(25.1)	...	....	10	10 (20.0)	9.7	9.7(39.4)	...	....
No. 54	9.6	7.3(16.9)	1.0	0(17.9)	0	0(17.9)	10	10 (20.0)	9.5	9.2(38.7)	...	....
No. 16	10	9.7(19.7)	8.5	6.0(34.2)	5.1	....	10	10 (20.0)	10	10 (40.0)	9.9	....
No. 22	10	9.6(19.6)	9.0	9.0(37.6)	8.3	8.0(53.9)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 24	10	9.5(19.5)	9.0	9.0(37.5)	8.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 4	10	10 (20.0)	9.4	8.7(38.1)	8.7	7.0(53.8)	10	10 (20.0)	10	9.8(39.8)	9.7	9.3(58.8)
No. 18	10	9.6(19.6)	9.0	9.0(37.6)	8.3	8.0(53.9)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 25	10	9.5(19.5)	9.0	9.0(37.5)	8.3	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 50^a	9.9	9.4(19.3)	8.9	8.7(36.9)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 15	10	9.7(19.7)	9.0	9.0(37.7)	8.3	8.0(54.0)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 26	10	9.5(19.5)	9.0	9.0(37.5)	8.7	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 27	10	9.4(19.4)	9.0	9.0(37.4)	8.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 6	10	10 (20.0)	9.3	9.0(38.3)	9.0	8.1(55.4)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 7	10	10 (20.0)	9.3	9.0(38.3)	8.8	8.1(55.2)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 19	10	9.7(19.7)	9.0	9.0(37.7)	8.5	8.0(54.2)	10	10 (20.0)	10	9.9(39.9)	9.9	9.9(59.7)
COMPUTED C_3S CONTENT 31 TO 40 PER CENT												
No. 30	9.0	6.0(15.0)	2.6	0(17.6)	0	0(17.6)	10	10 (20.0)	9.9	9.9(39.8)	9.2	....
No. 39A	8.3	2.2(10.5)	0	0(10.5)	0	0(10.5)	10	9.5(19.5)	8.7	....	...	....
No. 29	9.7	6.9(16.6)	3.0	1.2(20.8)	0	0(20.8)	10	10 (20.0)	10	10 (40.0)	10	....
No. 32	10	8.9(18.9)	5.0	2.5(26.4)	0	0(26.4)	10	10 (20.0)	10	10 (40.0)	10	....
No. 21	10	9.6(19.6)	9.0	8.3(36.9)	5.5	5.2(47.6)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 14	10	9.7(19.7)	8.9	8.7(37.3)	0.2	0(37.5)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 13	10	9.7(19.7)	9.0	9.0(37.7)	8.1	6.3(52.1)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 23	10	9.5(19.5)	9.0	9.0(37.3)	8.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 17	10	9.7(19.7)	9.0	9.0(37.7)	8.3	8.0(54.0)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
No. 51	10	9.8(19.8)	8.9	8.7(37.4)	...	....	10	10 (20.0)	10	10 (40.0)	...	....
No. 5B	10	10 (20.0)	9.4	8.7(38.1)	8.7	8.0(54.8)	10	10 (20.0)	10	10 (40.0)	10	10 (60.0)
COMPUTED C_3S CONTENT 21 TO 30 PER CENT												
No. 8^a	9.6	0.7(10.3)	0	0(10.3)	0	0(10.3)	9.8	7.5(17.3)	4.4	1.3(23.0)	0	0(23.0)
No. 41	10	9.5(19.5)	9.0	9.0(37.5)	8.2	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 43	10	9.5(19.5)	9.0	9.0(37.5)	9.0	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 45	10	9.9(19.9)	9.0	9.0(37.9)	9.0	....	10	10 (20.0)	10	10 (40.0)	10	....
COMPUTED C_3S CONTENT BELOW 21 PER CENT												
No. 37	9.0	2.3(11.3)	0	0(11.3)	0	0(11.3)	9.5	4.6(14.1)	1.7	0(15.8)	0	0(15.8)

[a] These cements were double burned.

[b] Values in parentheses are "durability factors" and are the sums of all the individual ratings up to and including the period indicated.

TABLE IX.—COEFFICIENTS OF LINEAR EQUATIONS REPRESENTING RELATION BETWEEN THEORETICAL COMPOUND COMPOSITION AND STRENGTH.

Equations based on tests of 57 cements of approximately constant MgO content and containing 1 per cent or less free CaO.

The strength in lb. per sq. in. to be expected from a given composition may be calculated by means of the constants given in the table. For example the strength of 2-in. cubes of 1:2.75 mix by weight, tested at 1 day equals 8.5 C_3S + 0.3 C_2S + 11.3 C_3A − 6.5 C_4AF. Therefore, the expected strength of a cement with 72 per cent C_3S, 7 per cent C_2S, 2 per cent C_3A and 15 per cent C_4AF equals $(72 \times 8.5) + (7 \times 0.3) + (2 \times 11.3) + (15 \times -6.5) = 540$ lb. per sq. in.

The calculated strength is not an exact prediction of the strength which will be obtained, and therefore the probable error should be computed. The square of the probable error equals the sum of the squares of the products obtained by multiplying the per cent of each compound by the probable error of the corresponding coefficient. In this case,

$(\text{P.E.})^2 = (72 \times 0.40)^2 + (7 \times 0.37)^2 + (2 \times 1.11)^2 + (15 \times 1.26)^2 = 1198$

or, P.E. = ± 35. The expected strength is thus found to be 540 ± 35 lb. per sq. in.

It should be understood that this and similar computations apply strictly only to the conditions involved in the series from which the equations were obtained. Applications to other ranges of composition, conditions of manufacture, and conditions of test, amount to extrapolation, and are subject to the errors often encountered when extrapolation is attempted.

Compound	Coefficient of Linear Equations at Age Indicated							
	28 days in Moist Room, then in Water							28 days Moist, then Air
	1 day	3 days	7 days	28 days	3 months	1 year	2 years	1 year
2-in. Plastic Mortar Cubes, 1:2.75 Mix by Weight								
C_3S	8.5±0.40	27.4±0.98	40.0±1.47	48.8±3.10	55.7±3.67	61.8±4.10	70.7±4.05	....
C_2S	0.3±0.37	−1.1±0.91	−5.1±1.36	19.1±2.88	62.9±3.41	80.6±3.81	82.2±4.13	....
C_3A	11.3±1.11	24.1±2.74	58.4±4.11	100.1±8.67	56.4±10.28	5.6±11.47	−12.5±11.43	....
C_4AF	−6.5±1.26	−9.8±3.12	−0.2±4.68	30.8±9.88	39.7±11.71	39.6±13.07	27.2±13.12	....
2-in. Plastic Mortar Cubes, 1:4.25 Mix by Weight								
C_3S	2.7±0.15	9.8±0.40	14.6±0.70	20.1±1.37	22.0±1.90	25.8±2.15	29.6±2.07	....
C_2S	0.1±0.14	0.1±0.37	−2.4±0.65	6.4±1.27	30.7±1.77	43.5±2.00	41.3±2.11	....
C_3A	2.9±0.43	8.0±1.11	24.9±1.96	46.2±3.83	29.2±5.33	−8.4±6.01	−8.5±5.85	....
C_4AF	−2.0±0.48	−5.9±1.27	−3.9±2.24	−0.6±4.36	1.1±6.07	−2.9±6.85	−4.7±6.71	....
3 by 6-in. Concrete Cylinders, 1:2.4:3.6 Mix by Weight								
C_3S	....	....	....	41.4±2.23	....	54.8±2.68	....	52.9±2.70
C_2S	....	....	....	7.6±2.07	....	42.4±2.49	....	38.8±2.51
C_3A	....	....	....	123.4±6.25	....	100.4±7.49	....	70.4±7.56
C_4AF	....	....	....	37.8±7.12	....	53.5±8.54	....	35.2±8.62
1:3 Standard Ottawa Sand Briquets								
C_3S	2.1±0.14	3.6±0.19	4.6±0.21	5.0±0.20	4.7±0.17	4.6±0.26	4.9±0.23	....
C_2S	0.3±0.13	0.8±0.18	1.3±0.19	3.8±0.18	6.1±0.16	6.4±0.25	6.1±0.24	....
C_3A	4.6±0.39	6.3±0.54	7.1±0.57	7.1±0.55	4.4±0.48	2.1±0.74	0.9±0.65	....
C_4AF	0.4±0.45	3.7±0.62	3.5±0.65	4.0±0.63	4.0±0.55	2.6±0.84	2.2±0.74	....

NOTE.—If it should be desired to express the relation between composition and strength or any other property in terms of the oxides, coefficients for such equations may be readily obtained. Thus if *a*, *b*, *c* and *d* are the coefficients of C_3S, C_2S, C_3A and C_4AF respectively in the equation for strength for a particular type of specimen and test period, then

$$\text{Strength} = (4.0710a - 3.0710b)\,\text{CaO} + (8.6024b - 7.6024a)\,\text{SiO}_2 + (1.0785b + 3.0432d - 1.4297a - 1.6920c)\,\text{Fe}_2\text{O}_3 + (5.0683b - 6.7187a + 2.6504c)\,\text{Al}_2\text{O}_3.$$

TABLE X.—LENGTH CHANGES OF 3 BY 3 BY 15-IN. MORTAR BARS.

Mix: 1:3.6 by weight.
Aggregate: Elgin sand graded 0 to No. 4 sieve.
Water content of mortar: 6.35 gal. per sack cement.

Duplicate bars made for each cement in paraffined paper-lined wood molds covered for 1 day with a glass plate to prevent loss of moisture. Upon removal from molds, 1 bar stored in moist room until 7 days old, then in air of laboratory at approximately 50 per cent relative humidity until 14 months old, then in water. The other bar stored in moist room until 90 days old, then in air of laboratory at 50 per cent relative humidity.

Length changes measured on top sides of bars by means of fulcrum plate strain gage between holes drilled in brass plugs inserted in fresh mortar at time of molding (all values represent the change in length of the bars after 1 day in molds).

Cement	Expansion (+) or Contraction (−) of 3 by 3 by 15-in. Mortar Bars at Age Indicated, per cent															
	Moist Cured 7 days, then in Air and in Water									Moist Cured 3 months, then in Air of Laboratory						
	Moist Cured, 7 days	In Air at 50 per cent Relative Humidity					In Water			Moist Cured		In Air at 50 per cent Relative Humidity				
		1 month	3 months	6 months	12 months	14 months	15 months	20 months	26 months	7 days	3 months	4 months	6 months	1 year	2 years	3 years
Computed C$_3$S Content Above 60 per cent																
	(+)	(−)	(−)	(−)	(−)	(−)	(−)	(−)	(−)	(+)	(+)	(−)	(−)	(−)	(−)	(−)
No. 104[a]	0.004	0.073	0.082	0.086	0.088	0.088	.....	.....	.....	0.004	0.015	0.068	0.076	0.076	.....	.....
No. 106[a]	0.008	0.058	0.081	0.085	.....	.....	.....	.....	.....	0.008	0.014	0.058	0.078	.....	.....	.....
No. 102[a]	0.010	0.063	0.083	0.084	0.085	0.085	0.049	.....	.....	0.009	0.024	0.060	0.068	0.071	.....	.....
No. 38A[a]	0.004	0.070	0.092	0.098	0.101	0.101	0.068	0.063	.....	0.004	0.009	0.075	0.093	0.099	.....	.....
No. 58[a]	0.002	0.061	0.083	0.087	0.094	0.094	0.060	0.054	0.051	0.003	0.011	0.064	0.081	0.093	0.096	.....
No. 103[a]	0.006	0.056	0.076	0.077	0.078	0.078	0.046	.....	.....	0.007	0.012	0.064	0.067	0.068	.....	.....
No. 42	0.003	0.034	0.062	0.068	0.074	0.074	0.044	0.042	0.040	0.004	0.006	0.041	0.055	0.063	0.067	.....
No. 108[a]	0.004	0.053	0.073	0.077	.....	.....	.....	.....	.....	0.003	0.009	0.039	0.061	.....	.....	.....
No. 62[a]	0.003	0.033	0.063	0.066	0.075	0.076	0.041	0.036	.....	0.005	0.008	0.031	0.046	0.060	.....	.....
Computed C$_3$S Content 51 to 60 per cent																
No. 100[a]	0.007	0.068	0.096	0.099	0.100	0.100	0.065	.....	.....	0.006	0.012	0.069	0.080	0.087	.....	.....
No. 105[a]	0.006	0.055	0.062	0.064	0.066	0.067	.....	.....	.....	0.005	0.019	0.047	0.062	0.062	.....	.....
No. 40[a]	0.004	0.055	0.068	0.075	0.080	0.081	0.050	0.045	0.043	0.001	0.006	0.058	0.076	0.083	0.087	.....
No. 101	0.007	0.053	0.076	0.079	0.082	0.082	0.051	.....	.....	0.006	0.010	0.063	0.075	0.079	.....	.....
No. 49	0.000	0.056	0.074	0.078	0.084	0.085	0.056	0.053	0.050	−0.001	0.004	0.063	0.069	0.079	0.082	.....
No. 9[a]	0.005	0.051	0.074	0.077	0.081	0.081	0.052	0.046	0.046	0.005	0.008	0.042	0.066	0.074	0.079	0.083
No. 60	0.012	0.039	0.064	0.065	0.068	0.070	0.040	0.033	.....	0.011	0.024	0.038	0.053	0.062	.....	.....
No. 59	0.002	0.043	0.066	0.069	0.074	0.075	0.048	0.042	0.039	0.002	0.007	0.040	0.061	0.074	0.079	.....
No 60A	0.005	0.043	0.072	0.080	0.084	0.085	0.055	0.050	.....	0.005	0.006	0.048	0.064	0.072	.....	.....
No. 10[a]	0.004	0.041	0.063	0.067	0.069	0.070	0.043	0.038	0.037	0.005	0.010	0.034	0.056	0.063	0.068	0.071
No. 44	0.003	0.039	0.056	0.061	0.067	0.068	0.041	0.037	0.034	0.004	0.007	0.027	0.051	0.062	0.064	.....
No. 5B Dupl.	0.005	0.031	0.060	0.066	0.074	0.075	0.046	0.039	0.038	0.005	0.010	0.036	0.055	0.060	0.071	.....
No. 11	0.005	0.041	0.063	0.067	0.071	0.072	0.043	0.041	0.040	0.006	0.010	0.031	0.051	0.060	0.066	0.069
No. 46	0.004	0.042	0.057	0.061	0.070	0.072	0.043	0.038	0.036	0.003	0.006	0.029	0.053	0.064	0.071	.....
No. 61	0.002	0.038	0.059	0.061	0.068	0.069	0.041	0.035	0.031	0.003	0.009	0.032	0.051	0.064	0.068	.....
Computed C$_3$S Content 41 to 50 per cent																
No. 1 Dupl.	0.003	0.063	0.085	0.088	0.092	0.092	0.060	0.053	0.050	0.003	0.008	0.065	0.074	0.083	0.085	0.087
No. 1	0.006	0.058	0.096	0.097	0.098	0.099	0.067	0.060	0.059	0.004	0.014	0.060	0.079	0.084	0.091	0.093
No. 2B	0.005	0.043[b]	0.097	0.102	0.104	0.106	0.073	0.066	0.067	0.004	0.010	0.065	0.082	0.087	0.092	0.095
No. 8 Dupl.	0.004	0.065	0.080	0.087	0.092	0.093	0.060	0.059	0.057	0.004	0.006	0.062	0.077	0.082	0.087	0.090
No. 2B Dupl.	0.006	0.058	0.080	0.087	0.093	0.093	0.064	0.059	0.057	0.005	0.012	0.052	0.075	0.085	0.088	.....
No. 57	0.011	0.054	0.084	0.086	0.090	0.091	0.058	0.051	.....	0.011	0.021	0.057	0.070	0.076	.....	.....
No. 47	0.006	0.065	0.079	0.085	0.088	0.088	0.060	0.054	0.052	0.007	0.012	0.064	0.080	0.085	0.087	.....
No. 48	0.000	0.063	0.083	0.088	0.094	0.095	0.065	0.061	0.058	0.000	0.004	0.063	0.072	0.084	0.087	.....
No. 12	0.003	0.060	0.076	0.081	0.084	0.085	0.057	0.053	0.052	0.003	0.008	0.054	0.064	0.068	0.071	0.074
No. 20	0.002	0.061	0.076	0.078	0.081	0.082	0.057	0.052	0.051	0.005	0.009	0.060	0.071	0.077	0.084	0.088

No. 34	0.005	0.062	0.075	0.083	0.089	0.089	0.063	0.060	0.058	0.003	0.005	0.060	0.078	0.085	0.091	0.092
No. 35	0.005	0.059	0.067	0.073	0.078	0.078	0.053	0.046	0.041	0.004	0.014	0.051	0.068	0.073	0.078	0.081
No. 3B	0.006	0.059	0.077	0.082	0.086	0.087	0.061	0.055	0.054	0.006	0.013	0.059	0.077	0.082	0.091	0.091
No. 28	0.005	0.060	0.076	0.080	0.085	0.085	0.056	0.052	0.052	0.006	0.011	0.054	0.070	0.080	0.082	0.084
No. 31	0.003	0.058	0.067	0.071	0.077	0.078	0.051	0.048	0.048	0.003	0.005	0.056	0.070	0.077	0.082	0.083
No. 53[a]	0.005	0.053	0.076	0.081	0.087	0.088	0.054	0.048	0.044	0.004	0.012	0.049	0.066	0.080	0.084	.....
No. 54	0.003	0.054	0.072	0.075	0.082	0.083	0.056	0.051	0.048	0.003	0.008	0.063	0.069	0.078	0.080	.....
No. 16	0.008	0.044	0.065	0.068	0.070	0.071	0.045	0.039	0.037	0.008	0.017	0.043	0.057	0.068	0.073	0.075
No. 22	0.003	0.058	0.074	0.077	0.081	0.082	0.058	0.054	0.053	0.003	0.008	0.052	0.067	0.074	0.081	0.083
No. 24	0.004	0.046	0.066	0.068	0.073	0.074	0.048	0.045	0.044	0.004	0.007	0.042	0.061	0.068	0.074	0.076
No. 4	0.003	0.056	0.079	0.084	0.086	0.087	0.055	0.053	0.052	0.004	0.010	0.047	0.070	0.076	0.085	0.085
No. 18	0.003	0.046	0.065	0.068	0.072	0.072	0.047	0.043	0.042	0.003	0.009	0.042	0.057	0.068	0.070	0.072
No. 25	0.004	0.048	0.066	0.069	0.074	0.075	0.050	0.047	0.047	0.003	0.005	0.045	0.061	0.067	0.072	0.074
No. 50[a]	0.000	0.037	0.057	0.061	0.068	0.068	0.040	0.035	0.031	0.001	0.007	0.029	0.043	0.059	0.061	.....
No. 15	0.001	0.048	0.064	0.070	0.072	0.073	0.047	0.044	0.043	0.001	0.005	0.051	0.062	0.072	0.077	0.079
No. 26	0.006	0.045	0.063	0.065	0.071	0.072	0.047	0.043	0.042	0.006	0.010	0.040	0.059	0.068	0.072	0.074
No. 27	0.004	0.049	0.060	0.064	0.071	0.071	0.045	0.041	0.040	0.004	0.009	0.033	0.050	0.061	0.065	0.068
No. 6	0.004	0.036	0.055	0.063	0.067	0.068	0.044	0.039	0.038	0.005	0.009	0.034	0.056	0.062	0.072	0.072
No. 7	0.006	0.037	0.056	0.063	0.065	0.068	0.044	0.039	0.038	0.007	0.011	0.033	0.056	0.067	0.073	0.075
No. 19	0.004	0.040	0.058	0.062	0.067	0.068	0.044	0.040	0.040	0.002	0.006	0.039	0.055	0.066	0.071	0.074
COMPUTED C_3S CONTENT 31 TO 40 PER CENT																
No. 30	0.005	0.065	0.080	0.083	0.087	0.088	0.060	0.055	0.055	0.004	0.009	0.059	0.070	0.076	0.079	0.080
No. 39A	0.006	0.064	0.078	0.085	0.091	0.092	0.065	0.062	.....	0.005	0.010	0.050	0.073	0.080	.....	.....
No. 29	0.005	0.066	0.077	0.082	0.088	0.088	0.062	0.059	0.056	0.003	0.008	0.055	0.069	0.078	0.081	0.083
No. 32	0.004	0.056	0.064	0.068	0.071	0.072	0.046	0.039	0.039	0.003	0.014	0.047	0.061	0.065	0.066	0.067
No. 21	0.003	0.053	0.068	0.072	0.076	0.077	0.050	0.046	0.046	0.004	0.007	0.058	0.064	0.072	0.078	0.079
No. 14	0.003	0.055	0.070	0.073	0.077	0.078	0.051	0.048	0.048	0.004	0.008	0.053	0.065	0.072	0.079	0.081
No. 13	0.004	0.056	0.065	0.069	0.073	0.074	0.050	0.046	0.046	0.003	0.007	0.056	0.064	0.068	0.073	0.077
No. 23	0.003	0.053	0.067	0.068	0.071	0.072	0.050	0.044	0.043	0.004	0.009	0.043	0.060	0.067	0.072	0.074
No. 17	0.003	0.045	0.062	0.064	0.067	0.067	0.043	0.040	0.038	0.003	0.009	0.039	0.058	0.063	0.068	0.070
No. 51	0.001	0.049	0.066	0.068	0.077	0.078	0.051	0.046	0.042	−0.001	0.007	0.038	0.055	0.066	0.071	.....
No. 5B	0.005	0.042	0.059	0.067	0.072	0.073	0.046	0.043	0.042	0.004	0.008	0.046	0.063	0.068	0.082	0.083
COMPUTED C_3S CONTENT 21 TO 30 PER CENT																
No. 8[a]	0.004	0.069	0.096	0.099	0.101	0.102	0.076	0.068	0.065	0.006	0.011	0.056	0.076	0.081	0.085	0.088
No. 41	0.004	0.054	0.066	0.073	0.078	0.079	0.052	0.047	0.046	0.006	0.010	0.039	0.062	0.069	0.073	0.076
No. 43	0.003	0.043	0.056	0.062	0.069	0.071	0.050	0.044	0.042	0.003	0.010	0.028	0.058	0.069	0.077	.....
No. 45	0.005	0.044	0.055	0.061	0.069	0.071	0.048	0.043	0.042	Broken in Handling						
COMPUTED C_3S CONTENT BELOW 21 PER CENT																
No. 37	0.005	0.065	0.085	0.091	0.098	0.099	0.073	0.068	0.067	0.004	0.011	0.047	0.074	0.083	0.089	.....
MgO SUBSTITUTED FOR CaO; C_3S DECREASES																
No. 31	0.003	0.058	0.067	0.071	0.077	0.078	0.051	0.048	0.048	0.003	0.005	0.056	0.070	0.077	0.082	0.083
No. 3B	0.007	0.058	0.076	0.081	0.084	0.085	0.060	0.054	0.053	0.006	0.013	0.058	0.076	0.080	0.089	0.088
No. 3B Dupl.	0.005	0.059	0.078	0.083	0.087	0.088	0.062	0.055	0.054	0.005	0.013	0.060	0.078	0.083	0.093	0.093
No. 32	0.004	0.056	0.064	0.068	0.071	0.072	0.046	0.039	0.039	0.003	0.014	0.047	0.061	0.065	0.066	0.067
No. 33	0.005	0.061	0.070	0.076	0.081	0.081	0.055	0.049	0.044	0.004	0.015	0.050	0.064	0.066	0.068	0.069
No. 55	0.005	0.056	0.081	0.086	0.094	0.095	0.063	0.050	0.041	0.006	0.031	0.046	0.060	0.067	0.064	.....
MgO ADDED; C_3S CONSTANT																
No. 34	0.005	0.062	0.075	0.083	0.089	0.089	0.063	0.060	0.058	0.003	0.005	0.060	0.078	0.085	0.091	0.092
No. 3B	0.007	0.058	0.076	0.081	0.084	0.085	0.060	0.054	0.053	0.006	0.013	0.058	0.076	0.080	0.089	0.088
No. 3B Dupl.	0.005	0.059	0.078	0.083	0.087	0.088	0.062	0.055	0.054	0.005	0.013	0.060	0.078	0.083	0.093	0.093
No. 35	0.005	0.059	0.067	0.073	0.078	0.078	0.053	0.046	0.041	0.004	0.014	0.051	0.068	0.073	0.078	0.081
No. 36	0.005	0.060	0.067	0.071	0.074	0.074	0.046	0.041	0.035	0.006	0.015	0.044	0.058	0.062	0.064	0.066
No. 56	0.004	0.053	0.071	0.075	0.077	0.078	0.043	0.027	0.019	0.005	0.021	0.050	0.057	0.064	0.061	.....

[a] These cements were double burned.

[b] Moist cured 14 days instead of 7 days.

TABLE XI.—TESTS ON EFFECT OF MgO IN CEMENT.

(a) *Composition of Cements:*

In general the SO_3 content of the clinker was close to 0 per cent and did not exceed one-tenth of 1 per cent. In grinding, gypsum was added to give an SO_3 content in the finished cement of 1.8 per cent.

Titanium is considered as TiO_2 since its compound in cement is not known.

For length changes of mortar bars and results of miscellaneous tests see Tables X and XII.

Cement	Oxide Analysis of Clinker							Computed Composition of Clinker							
								Cement Compounds					Uncombined Oxides		
	SiO_2	Fe_2O_3	Al_2O_3	Total CaO	MgO	TiO_2	Free CaO	C_3S	C_2S	C_3A	C_4AF	C_2F	MgO	TiO_2	Free CaO
MgO Substituted for CaO; C_3S Decreases															
No. 31	22.9	3.1	6.1	66.3	1.2	0.4	0.3	49	29	11	9	0	1.2	0.4	0.3
No. 3B	22.7	3.2	6.1	64.4	3.1	0.5	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 3B Dupl	22.7	3.2	6.1	64.4	3.1	0.5	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 32	23.3	2.9	5.8	62.9	4.7	0.3	0	36	40	10	9	0	4.7	0.3	0
No. 33	22.7	3.2	6.2	60.5	7.1	0.4	0	27	45	11	10	0	7.1	0.4	0
No. 55	23.0	3.3	6.3	57.2	9.7	0.5	0	11	58	11	10	0	9.7	0.5	0
MgO Added; C_3S Constant															
No. 34	23.4	3.1	6.3	65.5	1.4	0.4	0.2	41	36	11	9	0	1.4	0.4	0.2
No. 3B	22.7	3.2	6.1	64.4	3.1	0.4	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 3B Dupl	22.7	3.2	6.1	64.4	3.1	0.4	0.3	43	33	11	10	0	3.1	0.5	0.3
No. 35	22.3	3.1	6.2	63.2	4.9	0.4	0.2	41	33	11	9	0	4.9	0.4	0.2
No. 36	21.5	3.1	6.1	61.9	7.0	0.4	0.3	42	30	11	9	0	7.0	0.4	0.3
No. 56	20.2	3.1	6.0	60.4	9.9	0.5	0.1	47	23	11	9	0	9.9	0.5	0.1

(b) *Strength Tests:*

For further details of tests, see Tables II, III, V and VII.

Cement	Strength at Age Indicated, lb. per sq. in.													
	Stored in Moist Room				Stored in Moist Room 28 Days, Remainder as Indicated									
					In Water				In 2 per cent $MgSO_4$ Solution			In 2 per cent Na_2SO_4 Solution		
	1 day	3 days	7 days	28 days	3 months	1 year^a		2 years	3 months^a	1 year^a	2 years^a	3 months^a	1 year^a	2 years^a
MgO Substituted for CaO; C_3S Decreases 1:2.4:3.6 Mix, 3 by 6-in. concrete cylinders														
						Water	Air							
No. 31	....	....	....	4740	....	5800	**4970(86)**	....	....	**5560(96)**	....	....	**3600(62)**	....
No. 3B	....	....	....	4130	....	6040	**4920(81)**	....	....	**2890(48)**	....	....	**1510(25)**	....
No. 3B Dupl	....	....	....	3770	....	5880	**4480(76)**	....	....	**Disintegrated**	....	....	**3720(63)**	....
No. 32	....	....	....	4050	....	5670	**4830(85)**	....	....	**4660(82)**	....	....	**5930(105)**	....
No. 33	....	....	....	3650	....	5480	**4830(88)**	....	....	**1570(29)**	....	....	**3490(64)**	....
No. 55	....	....	....	3010	....	4900	**4280(87)**	....	....	**Disintegrated**	....	....	**2590(53)**	....
1:2.75 Mix, 2-in. plastic mortar cubes														
No. 31	580	1480	2400	4470	6080	5930		6280	5350(88)	Disintegrated	Disintegrated	6280(103)	Disintegrated	Disintegrated
No. 3B	460	1260	2010	4460	6440	6930		6710	6020(93)	Disintegrated	Disintegrated	5850(91)	Disintegrated	Disintegrated
No. 3B Dupl	460	1190	1900	4090	5760	6280		6120	5550(96)	Disintegrated	Disintegrated	5430(94)	Disintegrated	Disintegrated
No. 32	430	1170	1900	4480	5650	5970		6190	5130(91)	Disintegrated	Disintegrated	5270(93)	Disintegrated	Disintegrated
No. 33	270	740	1060	3660	5670	5370		5450	3910(69)	Disintegrated	Disintegrated	5170(91)	Disintegrated	Disintegrated
No. 55	120	260	410	1910	3740	3940		2690	3130(84)	Disintegrated	Disintegrated	3540(95)	Disintegrated	Disintegrated

1:4.25 Mix, 2-in. plastic mortar cubes

No. 31	160	430	900	1850	2490	2440	2300	1920(77)	Disintegrated	Disintegrated	2560(103)	Disintegrated	Disintegrated
No. 3B	130	500	880	1990	2920	3190	2900	2240(77)	Disintegrated	Disintegrated	2530(87)	Disintegrated	Disintegrated
No. 3B Dupl	130	470	760	1690	2470	2830	2890	2140(87)	Disintegrated	Disintegrated	2530(102)	Disintegrated	Disintegrated
No. 32	130	360	620	1580	2630	2850	2730	1880(71)	Disintegrated	Disintegrated	2600(99)	Disintegrated	Disintegrated
No. 33	90	250	400	1240	2510	2600	2330	1330(53)	Disintegrated	Disintegrated	2210(88)	Disintegrated	Disintegrated
No. 55	40	80	150	680	1700	1850	1420	1640(96)	Disintegrated	Disintegrated	1830(108)	Disintegrated	Disintegrated

1:3 Standard Ottawa sand briquets

No. 31	195	330	360	440	520	485	470	475(91)	105(22)	7(1)	565(109)	545(112)	570(121)
No. 3B	110	275	355	455	525	390	455	530(101)	Disintegrated	Disintegrated	500(95)	540(139)	195(43)
No. 3B Dupl	105	260	340	395	510	475	450	545(107)	Disintegrated	Disintegrated	585(115)	450(95)	160(36)
No. 32	180	295	330	430	505	480	435	480(95)	95(20)	30(7)	505(100)	500(104)	465(107)
No. 33	125	245	295	420	480	365	295	500(104)	Disintegrated	Disintegrated	520(108)	435(119)	360(122)
No. 55	65	135	180	410	475	230	175	495(104)	Disintegrated	Disintegrated	495(104)	Disintegrated	Disintegrated

MgO Added; C_3S Constant

1:2 4:3.6 Mix, 3 by 6-in. concrete cylinders

						Water Air							
No. 34	....	....	....	4500	....	5960 5530(93)	....	....	3650(61)	....	....	5380(90)	....
No. 3B	....	....	....	4130	....	6040 4920(81)	....	....	2890(48)	....	....	1510(25)	....
No. 3B Dupl	....	....	....	3770	....	5880 4480(76)	....	....	Disintegrated	....	....	3720(63)	....
No. 35	....	....	....	4120	....	5310 4870(92)	....	....	1630(31)	....	....	5700(107)	....
No. 36	....	....	....	3910	....	4860 5000(90)	....	....	1170(24)	....	....	5600(115)	....
No. 56	....	....	....	3920	....	5590 4900(88)	....	....	900(16)	....	....	4990(89)	....

1:2.75 Mix, 2-in. plastic mortar cubes

No. 34	450	1210	2020	4580	5840	6350	6120	5060(87)	Disintegrated	Disintegrated	6300(108)	Disintegrated	Disintegrated
No. 3B	460	1260	2010	4460	6440	6930	6710	6020(94)	Disintegrated	Disintegrated	5850(91)	Disintegrated	Disintegrated
No. 3B Dupl	460	1190	1900	4090	5760	6280	6120	5550(96)	Disintegrated	Disintegrated	5430(94)	Disintegrated	Disintegrated
No. 35	440	1220	1770	4020	5180	5550	5960	5350(103)	Disintegrated	Disintegrated	5480(106)	Disintegrated	Disintegrated
No. 36	450	1310	2320	4230	5460	5830	5320	4930(90)	Disintegrated	Disintegrated	5620(103)	Disintegrated	Disintegrated
No. 56	470	1360	2260	4080	4840	4420	2550	4970(103)	Disintegrated	Disintegrated	4900(101)	Disintegrated	Disintegrated

1:4.25 Mix, 2-in. plastic mortar cubes

No. 34	140	410	710	1780	2630	2750	2570	1710(65)	Disintegrated	Disintegrated	2510(95)	Disintegrated	Disintegrated
No. 3B	130	500	880	1990	2920	3190	2900	2240(77)	Disintegrated	Disintegrated	2530(87)	Disintegrated	Disintegrated
No. 3B Dupl	130	470	760	1690	2470	2830	2890	2140(87)	Disintegrated	Disintegrated	2530(102)	Disintegrated	Disintegrated
No. 35	130	410	770	1810	2240	2410	2310	1320(59)	Disintegrated	Disintegrated	1980(88)	Disintegrated	Disintegrated
No. 36	140	420	860	1810	2480	2270	2130	1210(49)	Disintegrated	Disintegrated	1950(79)	Disintegrated	Disintegrated
No. 56	150	430	720	1490	1930	1560	600	1460(76)	Disintegrated	Disintegrated	1550(80)	Disintegrated	Disintegrated

1:3 Standard Ottawa sand briquets

No. 34	205	335	380	460	475	540	490	495(104)	165(31)	13(3)	530(112)	555(103)	570(116)
No. 3B	110	275	355	455	525	390	455	530(101)	Disintegrated	Disintegrated	500(95)	540(139)	195(43)
No. 3B Dupl	105	260	340	395	510	475	450	545(107)	Disintegrated	Disintegrated	585(115)	450(95)	160(36)
No. 35	160	260	330	435	435	405	360	435(100)	15(4)	Disintegrated	480(110)	260(64)	Disintegrated
No. 36	175	275	350	435	450	355	285	380(84)	50(14)	Disintegrated	500(111)	265(75)	Disintegrated
No. 56	175	310	365	475	485	295	255	400(82)	Disintegrated	Disintegrated	495(102)	320(108)	Disintegrated

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[a] Values in parentheses are the strengths of specimens stored in air or sulfate solutions expressed as percentages of the strength of similar specimens stored in water and tested at the same age.

Table XI.—*Continued.*

(*c*) *Ratings of Specimens Stored in Sulfate Solutions:*

For further details see Tables II, IV, VI and VIII.

Cement	Ratings After Storage in Sulfate Solutions for Periods Indicated											
	In 2 per cent $MgSO_4$ Solution						In 2 per cent Na_2SO_4 Solution					
	6 months	1 year[b]	1½ years	2 years[b]	2½ years	3 years[b]	6 months	1 year[b]	1½ years	2 years[b]	2½ years	3 years[b]
MgO Substituted for CaO, C_3S Decreases												
1:2.4:3.6 Mix, 3 by 6-in. Concrete Cylinders												
No. 31	10	9.7(19.7)	9.0	8.5(37.2)	7.6	....	10	9.8(19.8)	9.2	9.0(38.0)	8.6	....
No. 3B	9.9	8.8(18.7)	6.4	3.9(29.0)	2.0	1.0(32.0)	9.9	8.1(18.0)	7.1	6.2(31.3)	6.2	6.0(43.5)
No. 3B Dupl.	9.5	8.7(18.2)	3.4	1.2(22.8)	0	0(22.8)	9.9	9.4(19.3)	7.7	6.4(33.4)	5.9	5.2(44.5)
No. 32	10	9.9(19.9)	9.1	8.5(37.5)	7.5	....	10	10 (20.0)	9.7	9.5(39.2)	9.3	....
No. 33	9.9	9.2(19.1)	8.7	6.9(34.7)	4.0	....	10	9.6(19.6)	8.5	7.7(35.8)	7.1	....
No. 55	10	7.5(17.5)	1.6	0(19.1)	0	0(19.1)	9.8	9.0(18.8)	8.3	5.6(32.7)	....	....
1:2.75 Mix, 2-in. Plastic Mortar Cubes												
No. 31	8.5	2.2(10.7)	0	0(10.7)	0	0(10.7)	9.3	3.7(13.0)	0	0(13.0)	0	0(13.0)
No. 3B	9.0	3.6(12.6)	0	0(12.6)	0	0(12.6)	8.5	0(8.5)	0	0(8.5)	0	0(8.5)
No. 3B Dupl.	8.8	3.8(12.6)	0	0(12.6)	0	0(12.6)	8.0	1.8(9.8)	0	0(9.8)	0	0(9.8)
No. 32	8.1	2.7(10.8)	0	0(10.8)	0	0(10.8)	9.4	4.0(13.4)	0	0(13.4)	0	0(13.4)
No. 33	6.2	0.8(7.0)	0	0(7.0)	0	0(7.0)	6.7	0.9(7.6)	0	0(7.6)	0	0(7.6)
No. 55	7.1	0.5(7.6)	0	0(7.6)	0	0(7.6)	7.0	0.1(7.1)	0	0(7.1)	0	0(7.1)
1:4.25 Mix, 2-in. Plastic Mortar Cubes												
No. 31	5.6	0(5.6)	0	0(5.6)	0	0(5.6)	5.8	0.6(6.4)	0	0(6.4)	0	0(6.4)
No. 3B	5.0	0(5.0)	0	0(5.0)	0	0(5.0)	4.0	0(4.0)	0	0(4.0)	0	0(4.0)
No. 3B Dupl.	4.8	0(4.8)	0	0(4.8)	0	0(4.8)	5.0	0(5.0)	0	0(5.0)	0	0(5.0)
No. 32	5.9	0(5.9)	0	0(5.9)	0	0(5.9)	8.8	2.1(10.9)	0	0(10.9)	0	0(10.9)
No. 33	4.9	0.4(5.3)	0	0(5.3)	0	0(5.3)	6.9	1.0(7.9)	0	0(7.9)	0	0(7.9)
No. 55	6.2	0(6.2)	0	0(6.2)	0	0(6.2)	6.5	0.5(7.0)	0	0(7.0)	0	0(7.0)
1:3 Standard Ottawa Sand Briquets												
No. 31	10	8.9(18.9)	5.5	2.3(26.7)	0	0(26.7)	10	10 (20.0)	10	10 (40.0)	10	....
No. 3B	9.8	7.0(16.8)	0	0(16.8)	0	0(16.8)	10	9.7(19.7)	8.2	4.8(32.7)	3.3	3.0(39.0)
No. 3B Dupl.	9.9	6.4(16.3)	3.0	0(19.3)	0	0(19.3)	10	9.4(19.4)	6.2	3.7(29.3)	2.0	1.5(32.8)
No. 32	10	8.9(18.9)	5.0	2.5(26.4)	0	0(26.4)	10	10 (20.0)	10	10 (40.0)	10	....
No. 33	8.8	2.7(11.5)	0	0(11.5)	0	0(11.5)	10	10 (20.0)	10	9.9(39.9)	8.0	....
No. 55	7.6	0(7.6)	0	0(7.6)	0	0(7.6)	9.4	2.5(11.9)	0	0(11.9)	0	0(11.9)

MgO Added; C_3S Constant

1:2.4:3.6 Mix, 3 by 6-in. Concrete Cylinders

No. 34	10	9.2(19.2)	8.9	8.2(36.3)	7.0	....	10	9.5(19.5)	9.2	8.8(37.5)	8.5	....
No. 3B	9.9	8.8(18.7)	6.4	3.9(29.0)	2.0	1.0(32.0)	9.9	8.1(18.0)	7.1	6.2(31.3)	6.2	6.0(43.5)
No. 3B Dupl.	9.5	8.7(18.2)	3.4	1.2(22.8)	0	0(22.8)	9.9	9.4(19.3)	7.7	6.4(33.4)	5.9	5.2(44.5)
No. 35	10	9.3(19.3)	8.9	7.2(35.4)	5.4	....	9.9	9.7(19.6)	9.3	8.9(37.8)	8.7	....
No. 36	9.9	8.9(18.8)	8.6	6.7(34.1)	4.3	....	9.9	9.5(19.4)	9.0	8.6(37.0)	7.9	....
No. 56	9.8	8.8(18.6)	2.0	0(20.6)	0	0(20.6)	9.8	9.2(19.0)	8.8	8.1(35.9)	...	....

1:2.75 Mix, 2-in. Plastic Mortar Cubes

No. 34	7.7	1.3(9.0)	0	0(9.0)	0	0(9.0)	8.2	0(8.2)	0	0(8.2)	0	0(8.2)
No. 3B	9.0	3.6(12.6)	0	0(12.6)	0	0(12.6)	8.5	0(8.5)	0	0(8.5)	0	0(8.5)
No. 3B Dupl.	8.8	3.8(12.6)	0	0(12.6)	0	0(12.6)	8.0	1.8(9.8)	0	0(9.8)	0	0(9.8)
No. 35	8.2	2.3(10.5)	0	0(10.5)	0	0(10.5)	8.3	0(8.3)	0	0(8.3)	0	0(8.3)
No. 36	8.1	2.0(10.1)	0	0(10.1)	0	0(10.1)	8.1	2.9(11.0)	0	0(11.0)	0	0(11.0)
No. 56	8.7	2.8(11.5)	0	0(11.5)	0	0(11.5)	8.2	1.5(9.7)	0.1	0(9.8)	0	0(9.8)

1:4.25 Mix, 2-in. Plastic Mortar Cubes

No. 34	5.7	0.5(6.2)	0	0(6.2)	0	0(6.2)	5.6	0.5(6.1)	0	0(6.1)	0	0(6.1)
No. 3B	5.0	0(5.0)	0	0(5.0)	0	0(5.0)	4.0	0(4.0)	0	0(4.0)	0	0(4.0)
No. 3B Dupl.	4.8	0(4.8)	0	0(4.8)	0	0(4.8)	5.0	0(5.0)	0	0(5.0)	0	0(5.0)
No. 35	5.3	0(5.3)	0	0(5.3)	0	0(5.3)	4.7	0.5(5.2)	0	0(5.2)	0	0(5.2)
No. 36	5.2	0.7(5.9)	0	0(5.9)	0	0(5.9)	3.4	0.3(3.7)	0	0(3.7)	0	0(3.7)
No. 56	5.0	0(5.0)	0	0(5.0)	0	0(5.0)	4.6	0(4.6)	0	0(4.6)	0	0(4.6)

1:3 Standard Ottawa Sand Briquets

No. 34	10	9.0(19.0)	5.2	3.0(27.2)	0.8	....	10	10 (20.0)	10	10 (40.0)	10	....
No. 3B	9.8	7.0(16.8)	0	0(16.8)	0	0(16.8)	10	9.7(19.7)	8.2	4.8(32.7)	3.3	3.0(39.0)
No. 3B Dupl.	9.9	6.4(16.3)	3.0	0(19.3)	0	0(19.3)	10	9.4(19.4)	6.2	3.7(29.3)	2.0	1.5(32.8)
No. 35	9.6	3.8(13.4)	0	0(13.4)	0	0(13.4)	10	9.2(19.2)	6.2	0.2(25.6)	0	0(25.6)
No. 36	9.5	3.9(13.4)	0	0(13.4)	0	0(13.4)	10	9.2(19.2)	5.5	0.1(24.8)	0	0(24.8)
No. 56	9.4	4.4(13.8)	0.4	0(14.2)	0	0(14.2)	10	9.7(19.7)	6.4	3.6(29.7)	...	....

[b] Values in parentheses are "durability factors" and are the sums of all the individual ratings up to and including the period indicated.

TABLE XII.—MISCELLANEOUS TESTS OF CEMENTS.

So far as possible, tests made in accordance with A.S.T.M. Standard Methods of Sampling and Testing Portland Cement (C 77 - 32).

Values for air analysis, water for normal consistency and soundness are for a single determination. Values for No. 325 fineness, turbidimeter surface areas and time of setting are the average of 2 determinations.

Fineness at 10, 20, 30 and 40 μ determined by interpolation from air separations at stack velocities corresponding to 11, 21, 31 and 44 μ, by Stokes' Law. Air analyzer surface areas calculated from the separate sizes according to the formula 190/*d*, where *d* = average particle size of fraction in microns.

Cement	Fineness						Surface Area, sq. cm. per g.		Water for Normal Consistency, per cent by weight	Soundness over Boiling Water	Time of Setting			
	Air Analysis, per cent by weight finer than size indicated				Per cent by weight finer than Sieve						Vicat Needle		Gillmore Needle	
											Initial	Final	Initial	Final
	10μ	20μ	30μ	40μ	No. 325	No. 200	Air-Analyzer	Wagner Turbidimeter			hr.:min.	hr.:min.	hr.:min.	hr.:min.
C_3S CONTENT ABOVE 60 PER CENT														
No. 104^a	26.2	41.1	56.9	66.7	78.5	87.7	1440	1690	24.0	OK	2:50	6:10	4:00	6:30
No. 106^a	28.5	44.8	61.3	70.5	81.8	87.5	1540	1940	22.5	OK	2:00	4:45	3:30	5:25
No. 102^a	34.2	47.4	59.0	68.0	80.3	87.5	1690	1960	22.0	OK	1:35	4:20	2:30	5:20
No. 38A^a	23.0	38.8	53.4	62.6	74.5	87.4	1330	1480	22.0	OK	3:10	7:15	5:00	8:00
No. 58^a	24.5	39.8	55.0	64.1	75.1	87.6	1380	1570	21.0	OK	2:15	7:00	4:15	7:30
No. 103^a	29.6	47.2	63.4	75.0	83.3	87.7	1590	1870	22.0	OK	3:20	7:10	4:20	7:45
No. 42	25.5	40.7	57.8	67.3	77.3	87.4	1430	1570	21.5	OK	3:40	7:30	5:10	8:05
No. 108	30.8	48.0	61.8	70.3	80.8	87.5	1610	1785	21.5	OK	4:30	8:05	5:40	8:30
No. 62^a	25.6	42.2	48.0	63.5	75.9	87.7	1410	1550	21.5	OK	4:30	9:00	6:00	9:50
C_3S CONTENT 51 TO 60 PER CENT														
No. 100^a	29.9	43.3	57.7	66.8	79.0	87.7	1550	1690	22.0	OK	2:00	4:30	3:10	6:00
No. 105^a	27.0	44.3	59.1	68.2	79.5	87.9	1490	1720	22.0	OK	3:10	6:55	4:45	7:15
No. 40^a	25.2	40.2	55.5	60.8	74.0	87.8	1400	1390	21.5	OK	4:30	7:35	5:35	8:15
No. 101	28.4	43.4	58.6	68.0	79.9	87.7	1520	1710	21.5	OK	2:35	5:20	3:50	6:40
No. 49	26.7	41.8	56.3	63.6	74.8	87.4	1450	1515	21.5	OK	4:00	7:20	5:00	8:00
No. 9^a	25.5	40.1	53.6	60.1	72.9	87.6	1400	1465	21.0	OK	4:45	7:25	5:35	8:10
No. 60	29.7	53.2	55.7	62.6	73.3	87.5	1580	1710	20.5	OK	1:30	4:30	2:30	5:10
No. 59	25.2	40.2	54.6	62.7	73.9	87.4	1400	1525	21.0	OK	3:50	7:10	5:30	7:55
No. 60A	26.5	41.6	56.1	64.9	75.7	87.4	1450	1555	22.0	OK	4:30	8:20	6:00	9:10
No. 10^a	25.7	39.3	53.0	57.4	73.6	88.2	1390	1480	20.5	OK	4:50	7:30	6:00	8:20
No. 44	25.1	39.4	55.0	63.8	75.2	87.8	1400	1475	21.0	OK	5:15	8:35	6:10	8:45
No. 5B Dupl.	26.6	42.5	55.3	62.9	74.5	87.9	1440	1595	21.0	OK	4:10	7:40	4:55	8:25
No. 11	26.6	40.4	54.1	59.3	73.8	87.9	1420	1485	20.5	OK	4:50	8:35	5:45	9:25
No. 46	24.7	38.6	54.3	63.7	75.3	88.1	1380	1465	21.0	OK	4:05	9:05	5:30	9:55
No. 61	26.9	40.7	53.7	61.5	72.3	87.4	1440	1525	21.0	OK	4:00	7:50	5:20	8:40
C_3S CONTENT 41 TO 50 PER CENT														
No. 1 Dupl.	25.6	40.7	54.3	64.1	75.9	87.8	1410	1645	22.0	OK	3:30	6:15	4:15	7:15
No. 1	28.8	42.3	57.3	63.6	76.5	87.3	1510	1695	21.5	OK	2:10	5:05	3:15	5:35
No. 2B	23.5	39.9	55.6	62.2	75.8	87.2	1350	1370	21.5	OK	2:35	5:15	3:35	5:35
No. 8 Dupl.	27.1	40.1	53.4	62.7	74.8	87.7	1440	1575	21.5	OK	3:50	7:25	5:15	7:45
No. 2B Dupl.	26.2	40.8	54.4	63.1	75.4	87.3	1420	1590	21.0	OK	2:35	6:35	3:50	6:45
No. 57	30.7	42.7	54.6	62.5	74.3	87.1	1550	1790	20.5	OK	1:20	3:20	2:15	4:05
No. 47	24.5	41.5	55.2	61.3	75.6	87.5	1390	1590	21.0	OK	2:20	5:30	3:20	6:35
No. 48	26.6	40.7	53.4	62.5	74.4	87.9	1430	1515	22.0	OK	3:05	6:45	4:35	7:15
No. 12	25.6	39.7	54.4	59.4	74.0	88.2	1400	1470	21.5	OK	4:25	7:50	5:15	8:20
No. 20	26.9	41.4	55.4	64.2	77.9	88.3	1460	1590	21.5	OK	4:00	7:10	4:40	7:35
No. 34	26.8	39.9	54.4	62.7	73.7	87.3	1430	1550	20.5	OK	3:50	7:40	4:50	7:50

No. 35	25.4	39.2	53.5	62.6	74.1	87.4	1390	1510	22.5	OK	3:45	7:55	4:50	7:55
No. 3B	26.2	42.2	55.2	64.4	76.5	87.4	1440	1635	21.0	OK	2:55	6:35	5:20	7:40
No. 3B Dupl	26.2	40.7	52.3	61.5	75.5	87.2	1420	1610	21.0	OK	3:10	6:05	4:05	6:15
No. 28	26.8	42.4	56.5	62.3	76.3	88.7	1450	1665	22.0	OK	4:20	8:15	6:15	8:35
No. 31	26.3	41.1	54.3	64.0	75.0	87.4	1430	1525	21.0	OK	3:35	6:20	4:45	7:10
No. 53[a]	23.5	34.3	53.5	61.6	73.0	87.6	1320	1375	22.0	OK	3:00	6:35	4:05	7:00
No. 54	27.2	41.3	55.3	60.9	73.3	87.8	1450	1600	21.0	OK	2:15	6:00	3:35	6:35
No. 16	26.9	40.2	52.8	60.0	75.1	87.6	1430	1625	21.0	OK	2:35	5:20	3:30	5:50
No. 22	26.7	41.2	53.6	62.6	75.6	87.2	1440	1605	21.0	OK	4:40	8:20	6:35	9:05
No. 24	26.9	42.1	55.5	63.3	75.7	87.5	1450	1675	21.0	OK	5:30	8:25	6:25	9:35
No. 4	28.4	40.9	54.0	63.7	76.3	87.6	1480	1530	21.75	OK	4:15	8:10	5:30	8:35
No. 18	26.9	40.1	52.9	61.5	74.8	87.3	1430	1575	21.0	OK	5:05	7:10	6:05	8:10
No. 25	27.0	41.8	54.4	63.2	75.5	87.7	1450	1650	21.5	OK	4:35	9:40	6:00	9:55
No. 50[a]	23.3	37.3	50.5	59.1	73.0	87.9	1320	1330	21.0	OK	4:55	8:30	5:55	9:05
No. 15	25.7	45.7	54.0	63.0	76.1	87.5	1440	1580	21.0	OK	5:20	8:00	6:00	8:40
No. 26	27.2	41.5	53.4	62.2	74.7	88.2	1450	1635	20.5	OK	4:50	9:40	6:00	10:00
No. 27	27.4	42.9	55.0	62.7	73.6	87.2	1460	1535	20.5	OK	4:45	9:45	5:50	10:00
No. 6	26.6	40.7	51.7	61.1	74.0	87.2	1420	1530	21.0	OK	4:30	9:20	6:25	10:15
No. 7	25.9	40.6	51.5	61.0	74.8	87.6	1410	1370	20.0	OK	3:30	9:55	6:25	10:35
No. 19	26.6	40.5	54.9	61.0	76.2	87.2	1430	1590	20.5	OK	5:35	8:35	6:10	9:35

C_3S CONTENT 31 TO 40 PER CENT

No. 30	25.2	40.5	55.2	60.9	74.1	87.4	1400	1575	22.0	OK	4:05	8:15	6:30	8:50
No. 39A	26.3	40.8	53.1	61.0	73.0	87.5	1420	1610	21.5	OK	3:35	7:30	4:55	8:10
No. 29	25.5	41.2	55.3	61.3	74.5	87.3	1410	1580	22.0	OK	4:25	8:50	6:10	9:15
No. 32	25.7	40.1	52.8	62.2	74.7	87.6	1400	1570	21.0	OK	4:00	7:45	5:00	7:50
No. 21	25.7	40.5	53.3	63.3	76.5	87.2	1410	1555	21.0	OK	4:30	8:05	5:50	9:05
No. 14	26.6	40.8	54.5	62.2	74.7	87.6	1430	1595	21.5	OK	4:30	7:35	5:30	7:55
No. 13	25.9	40.7	55.0	59.9	74.5	87.0	1420	1520	21.5	OK	4:55	8:50	5:55	9:35
No. 23	26.7	41.7	56.1	63.9	75.8	87.1	1450	1605	21.0	OK	4:50	9:05	6:55	10:00
No. 17	23.9	40.4	54.7	60.7	75.2	87.2	1360	1540	21.0	OK	5:25	8:05	6:20	9:00
No. 51	26.8	40.5	50.9	60.2	73.1	87.3	1430	1565	21.0	OK	5:10	8:35	6:00	9:25
No. 5B	25.4	40.6	52.3	61.6	73.6	87.8	1400	1380	21.0	OK	3:30	8:20	5:10	8:45

C_3S CONTENT 21 TO 30 PER CENT

No. 8[a]	26.2	40.3	54.1	60.5	74.2	88.4	1420	1660	21.5	OK	2:20	6:00	3:55	6:25
No. 41	28.5	40.2	53.4	61.8	73.2	87.2	1470	1625	20.5	OK	3:20	7:35	5:05	8:05
No. 43	27.4	40.2	52.4	61.1	73.0	87.1	1440	1565	20.0	OK	4:05	8:25	5:45	9:20
No. 45	26.0	48.9	53.4	62.0	73.6	87.3	1450	1590	20.0	OK	5:00	10:25	6:25	11:20

C_3S CONTENT BELOW 21 PER CENT

No. 37	26.7	40.7	53.7	63.3	74.0	87.7	1440	1605	21.5	OK	3:00	7:10	5:00	8:00

MgO SUBSTITUTED FOR CaO; C_3S DECREASES

No. 31	26.3	41.1	54.3	64.0	75.0	87.4	1430	1525	21.0	OK	3:35	6:20	4:45	7:10
No. 3B	26.2	42.2	55.2	64.4	76.5	87.4	1440	1635	21.0	OK	2:55	6:35	5:20	7:40
No. 3B Dupl	26.2	40.7	52.3	61.5	75.5	87.2	1420	1610	21.0	OK	3:10	6:05	4:05	6:15
No. 32	25.7	40.1	52.8	62.2	74.7	87.6	1400	1570	21.0	OK	4:00	7:45	5:00	7:50
No. 33	27.7	40.3	52.8	62.2	73.7	87.5	1460	1625	21.0	OK	4:05	8:00	4:55	8:05
No. 55	27.2	40.7	51.6	59.6	71.0	87.9	1430	1610	22.0	OK	3:50	7:40	5:50	8:40

MgO ADDED; C_3S CONSTANT

No. 34	26.8	39.9	54.4	62.7	73.7	87.3	1430	1550	20.5	OK	3:50	7:40	4:50	7:50
No. 3B	26.2	42.2	55.2	64.4	76.5	87.4	1440	1635	21.0	OK	2:55	6:35	5:20	7:40
No. 3B Dupl	26.2	40.7	52.3	61.5	75.5	87.2	1420	1610	21.0	OK	3:10	6:05	4:05	6:15
No. 35	25.4	39.2	53.5	62.6	74.1	87.4	1390	1510	22.5	OK	3:45	7:55	4:50	7:55
No. 36	26.9	40.6	55.0	64.7	75.8	88.2	1450	1560	22.0	OK	3:45	7:35	4:45	7:35
No. 56	27.0	41.3	56.1	63.8	74.5	88.1	1450	1520	21.0	OK	3:35	7:20	5:00	8:10

[a] These cements were double burned.

DISCUSSION

Mr. R. H. Bogue.[1]—It is of interest to understand why the concept of computed compound composition of cements has not been generally applied in the cement industry until relatively recently. It was not until Rankin and Wright, at the Geophysical Laboratory, made studies of the phase-equilibria of the lime-alumina-silica system that data were gathered which permitted an understanding of what the composition must be of systems similar to portland cement. One reason is the natural complexity of the material; another the finely divided state of the crystalline material in the cement clinker and the impossibility of taking a piece of that clinker, shaving off a particular crystal and analyzing it to find out what it is. One could examine those materials under the microscope and determine their optical properties, but there was no means of arriving at a knowledge of their composition except by the unsatisfactory method of attempting to make a material which would have those same optical properties.

But that aspect was changed with the advent of the study by Rankin and Wright. Their study of the lime - alumina-silica system was and remains the classic upon which scores of investigations on other systems have been based. In that study they showed that tricalcium silicate, dicalcium silicate and tricalcium aluminate are the stable phases in the region of portland cement. They used the microscope as a basis for identification, but even then there was considerable dissension because, for example, tricalcium silicate could not be obtained by crystallization from a melt of the two components. There were other differences of opinion brought out by German chemists. Janecke did not believe that tricalcium silicate could exist in portland cement, even though it were defined by Rankin as a stable phase in these three components of the lime - alumina-silica system. He believed the principal compound of cement to be $8CaO \cdot Al_2O_3 \cdot 2SiO_2$. Others believed it to be a solid solution of aluminates in silicates and still others a solid solution of lime in dicalcium silicate.

The work started by Rankin and continued by Bates, at the Bureau of Standards, and later by our own staff has demonstrated conclusively that the "alite" or principal compound of portland cement is tricalcium silicate. Following a correspondence with Janecke, I sent him some of our material, and about a year later he graciously published a paper in which he agreed with our conclusions and admitted that tricalcium silicate is the principal compound in portland cement. Shortly thereafter, Guttman and Gille, leading cement chemists of Germany, said in a paper, "At last the 50-year-old problem of the constitution of cement, as to what is alite, is settled."

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[1] Research Director of the Portland Cement Assn. Fellowship at the National Bureau of Standards, Washington, D. C.

The criteria upon which cement compounds may be computed are based on evidence obtained by microscopic, X-ray and chemical methods. I might say a word about the adequacy, together with the failings, of our newest tool, the X-ray method. Unfortunately, we cannot say with certainty that a failure to obtain an X-ray diffraction pattern of a compound means that there is none of that particular compound present. If we fail, for example, to get the pattern of tricalcium aluminate from a cement, we cannot say it is not there, because it takes a certin amount of material to give a pattern strong enough to be identified in conjunction with the other materials present. That is its failing, but its tremendous advantage, an advantage which it has over the microscopic method, is that once you do obtain the pattern, there is no uncertainty; you know that the compound is present in an amount equal to or greater than a certain established amount. Some compounds, like lime, can be detected when present in amounts of 2 or 3 per cent; tricalcium aluminate requires about 6 per cent, and some other compounds require even more.

I think it is of particular interest that, in these data which Mr. Gonnerman has presented, there is such a beautiful confirmation of our fundamental concept that the properties of a cement must be a function of the compounds present rather than of the components, just as the properties of a salt solution are functions of the sodium chloride present and not of the sodium or chlorine as such.

The chief criticism that has been directed against the application of the concept of the compound composition to commercial clinkers is that there are occasional commercial cements whose properties are not in line with those anticipated by the computed composition. That is true. Those cements are of great interest to us. Those are the cements that our research group must be especially concerned with; we must find out why they do not fall in line. There is something about them that we do not know, something perhaps about their composition or their thermal history, or their later treatment that we have yet to find out. The cements used in the present investigation were made under rigidly controlled conditions of composition, raw fineness, thermal treatment, and cement fineness. These conditions are not similarly controlled in commercial practice; the variables involved, such as the temperature, the duration of the heating period, the rate of cooling, the impurities in the materials, etc., are not standardized, so the history of commercial cements, especially the thermal history, is not known in the degree to which it is known with the cements reported on by Mr. Gonnerman.

But in spite of these unsolved problems which still remain, and there are many, studies as reported here and elsewhere on the relationship betwen computed compound constitution and heat of hydration, sulfate resistance, strength, durability, etc., have been so definite in their results that in my opinion the industry is now justified in looking to the prediction of behavior in terms of the fundamental concept of compound composition.

MR. BRADLEY DEWEY.[1]—May I ask Mr. Bogue whether the evidence is in favor of the assumption that the clinker in burning is at a high enough temperature for a long enough time so that phase rule equilibria are reached.

MR. BOGUE.—All the evidence that we have, which includes chemical, microscopic and X-ray, is to the effect that equilibrium usually is very nearly reached, not quite, but very nearly reached. Moreover, the free lime test serves as a measure of the degree to which combination has been effected and is thus considered in the computation of the compounds actually produced.

MR. M. A. SWAYZE.[2]—The X-ray pattern referred to by Mr. Bogue, I believe, was determined by using the pure compound. If mixtures of this pure compound with other materials fails to show the characteristic lines when the compound is present in less than 6 or 7 per cent, then I believe we are justified in assuming that alumina present in commercial clinker in sufficient amounts to give a theoretical 6 or 7 per cent of tricalcium aluminate is actually present in that form in the clinker, providing, of course, that the typical lines for tricalcium aluminate appear when the theoretical percentage of tricalcium aluminate exceeds this amount. I would like to ask Mr. Bogue if any X-ray patterns have been taken of dilutions of pure tricalcium aluminate with other materials to check the point where the lines for tricalcium aluminate disappear.

MR. BOGUE.—Mr. Swayze is correct in thinking that we have made X-ray diffraction patterns of mixtures of the pure tricalcium aluminate with the other cement compounds. My statement of the required minimum percentage of tricalcium aluminate to give the

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[1] President, Dewey & Almy Chemical Co., Cambridge, Mass.

[2] Chief Chemist, Lone Star Cement Co. New York, Inc., Hudson, N. Y.

X-ray diffraction pattern of that compound was based on the results of tests with these mixtures of the pure compounds.

Our studies have not yet advanced to a point where we can say with certainty the nature of the alkali compounds in portland cement. The systems $CaO\text{-}Na_2O\text{-}Al_2O_3$ and $CaO\text{-}K_2O\text{-}Al_2O_3$ have been investigated and the compounds $8CaO \cdot Na_2O \cdot 3Al_2O_3$ and $K_2O \cdot Al_2O_3$ were found to be stable in the regions of portland cement. But the stability of those compounds has not yet been determined in the presence of the other cement compounds.

MR. P. S. ROLLER[1] (*by letter*).—Among the interesting relationships found in this comprehensive work, the frequent, rather abrupt change of properties with increase in computed C_3A attracts attention. Ordinarily one would expect a rather uniform change proportional to the amount of C_3A.

Two possibilities suggest themselves as the cause of the abrupt transition. At a low alumina content, an appreciable portion of the alumina in cement may not unite to form C_3A; furthermore, the occurrence and physical structure of the C_3A (also of the other compounds) may be different. It is highly probable that the alkalies in cement are combined with alumina in a binary or ternary compound. The combination would absorb 1 or 2 per cent of the alumina, which otherwise would go to form C_3A. (1 per cent of Al_2O_3 is equivalent to 2.7 per cent C_3A.)

Tricalcium aluminate in small amount might exist, not independently but isomorphously, with C_3S. This possibility was suggested by P. H. Bates on the basis of observations by Anderson and Lee[2] of isomorphism of C_3S with various compounds. I have subsequently found that Schwiete and Büssem[3] came to the conclusion from sensitive X-ray evidence that C_3A may be taken up isomorphously by C_3S to the extent of 4.5 per cent and possibly more.

It is questionable whether one is ever justified in ignoring the physical mode of occurrence of compounds, even if one is sure that the compounds are all there as supposed. Physical structure is best studied microscopically or by X-ray. Chemical composition, however, may be highly indicative in this respect. Empirical composition ratios, while reflecting compound composition, may also reflect changes in compound structure and may do so in a better way than the calculated compound composition.

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[1] Associate Chemist, U. S. Bureau of Mines, Nonmetallic Minerals Experiment Station, New Brunswick, N. J.

[2] O. Andersen and H. C. Lee, "Properties of Tricalcium Silicate from Basic Open-Hearth Steel Slags," *Journal*, Washington Academy of Sciences, Vol. 23, p. 338 (1933).

[3] H. E. Schwiete and W. Büssem, "Investigation of a Deposit-zone in a Rotary Kiln. The Alite Problem," *Tonindustrie Zeitung*, Vol. 56, p. 801 (1932).

From this point of view, it would be desirable to attempt to correlate the various properties studied by Gonnerman with empirical ratios such as SiO_2 to $Al_2O_3 + Fe_2O_3$, SiO_2 to Al_2O_3, Al_2O_3 to Fe_2O_3, etc., as well as with computed compound composition. The writer has recently obtained evidence that such ratios are more significant than computed compound composition in regard to susceptibility to reversion of set on storage. Presumably correlation of composition ratios has been attempted by Gonnerman, but I believe the results might actually be given because of their interest.

MR. L. B. MILLER[1] (*by letter*).—It is most encouraging to see the fundamental importance and practical value of phase rule studies of portland cement so clearly appreciated and so neatly applied as has been done in these studies of cements carried out by the Portland Cement Association at their Chicago laboratory and at the Bureau of Standards.

The important crystalline mineral constituents of portland cement have been discovered by means of phase rule investigations and described in detail through optical and X-ray studies. As Mr. Bogue has indicated, well-burned cement clinker represents a system which has reached substantial equilibrium and can therefore be described in terms of appropriate phase rule diagrams. There is one constituent of cement clinker not ordinarily included in a list of clinker constituents but which may have some importance. That constituent is glass—the uncrystallized remnant remaining as equilibrium is approached in the firing of the clinker.

It is not difficult to demonstrate the presence of such glass in cement clinker. It first came to my attention while attempting to check the optical constants of dicalcium silicate in clinker with published data on these constants. The result which I obtained for an index of refraction of this mineral constituent was entirely different from any published value. Closer observation revealed a film of glass surrounding the dicalcium silicate crystals and it was the index of refraction of this glass which was being measured. Suitable samples of clinker, not too finely ground, placed in a liquid whose index of refraction differs appreciably from that of the glass and of the crystalline clinker constituents and viewed under a high-power microscope readily reveal the presence of glass surrounding the crystalline mineral constituents. The presence of glass may be inferred from other data. It is well known that reactions in the solid state proceed very slowly. The presence of some liquid phase greatly accelerates such reactions and it is through the agency of some portion of liquid phase in the clinker that reactions in the cement kiln proceed to substantial equilibrium in so short a time. The fact that cement raw materials "ball up" during the firing process to form clinker is direct evidence of the formation of some liquid phase which remains in part as a glass upon cooling the clinker.

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[1] Research Chemist, Johns-Manville Research Laboratories, Manville, N. J.

The amount of glass present in cement clinker varies considerably. The amount is chiefly dependent on the composition of the clinker and its thermal history. It is exceedingly difficult to estimate even approximately how much glass is present in a given sample of clinker because it is dispersed between and around the crystalline constituents of the clinker. It is probably safe to say that there are several per cent of glass present in even a well-crystallized clinker. It seems probable that the glass may often vary considerably from the composition of the crystalline portion of the clinker. As clinker formation proceeds towards equilibrium the chief components of the cement will form those crystalline constituents which the phase rule indicates are stable and "insoluble" under existing conditions. The glass will probably consist of the chief components plus most of the "impurities" present in small amounts. This is especially to be expected where such "impurities" form easily fusible compositions with the major components: the alkali oxides are an example.

In studying clinker constituents by X-ray methods it is necessary for a crystalline constituent to be present beyond a certain threshold value, as Mr. Bogue has pointed out, before a distinct spectrum of the constituent makes its appearance. It is well known that glass yields no spectrum since it is non-crystalline in character. It may well be that the glass, by holding portions of certain of the components in a non-crystalline state, may increase the apparent threshold value for a given constituent where the percentage of such constituent present in the clinker is based on a calculation which assumes a clinker 100 per cent crystalline in character.

It has been indicated that the glass found in cement clinker normally is found between and around the crystalline constituents in the form of a thin coating like a matrix in which crystal formation has occurred. It seems fairly obvious that this glass, which serves in some measure to bind together the crystals composing the major portion of the clinker, should have some effect upon certain physical properties of the clinker such as the ease of pulverizing. It is well known that methods of quenching the hot clinker alter its grindibility; these would likewise alter the amount and character of the glass. Likewise, it seems fairly obvious that the hydraulic properties of the coarser fraction of cement should be influenced by the presence of glass since in general the coarser particles in cement consist of aggregates of a number of small glass-coated crystals. (The finest portion of a finished cement, on the other hand, consists usually of fragments of individual crystals.) Since the glass would in a measure protect the crystalline constituents from the water for a time at least, the hydraulic properties of the glass itself would be exhibited to some extent in the coarser fractions of cement rather than the hydraulic properties of the crystalline constituents only.

It is a personal belief based upon considerable observation that the glass constituent of cement clinker is in some cases of importance, quantitatively, and that a complete exposition of the properties of portland cement must include consideration of this constituent.

MR. H. F. GONNERMAN[1] (*author's closure by letter*).—The studies presented are based upon current ideas concerning the chemical nature of portland cement clinker. There are other things still to be learned, such as the influence of methods of burning and cooling mentioned by Mr. Bogue, the influence of minor constituents discussed by Mr. Roller, and the influence of glass suggested by Mr. Miller. All of these questions will eventually have to be considered. The degree of success which has been attained with our present knowledge warrants the belief that as new information on the chemical nature of portland-cement clinker is developed, there will be a corresponding advance in our understanding of the causes of differences in behavior of portland cements.

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[1] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

A SUSPENSION TURBIDIMETER FOR DETERMINATION OF SPECIFIC SURFACE OF GRANULAR MATERIALS

By Alexander Klein[1]

Synopsis

This paper describes a portable apparatus for the rapid determination of the surface area of portland cement and other granular materials, in which determination the turbidity of a suspension of the material in castor oil is measured. The method does not involve Stokes' law which is the basis of sedimentation methods, but consists in determining the mean effective cross-sectional area of the suspended material under static conditions, through the use of photoelectric equipment. Hence, no consideration of time or velocity is involved in the determinations, no standard samples are required, and the method is independent of the physical characteristics of the suspending medium. A theoretical constant required for the analysis of a given material is determined from an observation on a sieved sample of the material itself. A high degree of reproducibility is attained, and the method appears to furnish an accurate measure of the relative fineness of various materials.

Comparisons are made between test results obtained by means of this apparatus and by means of apparatus employing the principle of sedimentation.

Introduction

For lack of more refined methods, the fineness of portland cement and other granular materials has until recently been measured almost entirely by means of sieves. With the advent of finer grinding and increased percentages of sub-sieve particle sizes, the sieve analysis has proven inadequate as a measure of fineness. Recently, air separation and sedimentation methods have been found useful in the determination of particle-size distribution of material, the data being employed further to calculate the surface area of a unit weight of the material, or the specific surface.

It appears that surface area is, for practical purposes, a satisfactory measure of the fineness of cement. Recent studies[2] of the hydration of cement indicate that surface area adequately defines the fineness of cements for most purposes. While there is at present no absolute measure of surface area, such an absolute measure is not of vital importance provided some reasonable standard is employed. The accuracy to which specific surface must be measured will depend upon the purpose for which the measure is used. Where specific surface is employed to study the effect of fineness upon compressive strength or heat of hydration of concrete, it is found that the rate of hydration is at early ages roughly proportional to the fineness as expressed by surface area; hence the percentage of accuracy required in measuring the fineness should not be less than that required in measuring compressive strength or heat of hydration. Where the specific surface is employed in comparative grinding studies, it is found that as small a difference as 1 per cent in the total specific surface is of significance. While a like degree of accuracy is desired for research work or plant control, somewhat lower accuracy can be tolerated in acceptance tests on cement.

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[1] Assistant Research Engineer, Engineering Materials Laboratory, University of California, Berkeley, Calif.

[2] R. E. Davis, R. W. Carlson, G. E. Troxell and J. W. Kelly, "Cement Investigations for the Hoover Dam," *Proceedings* Am. Concrete Inst., Vol. 29, p. 413 (1933); "Cement Investigations for Boulder Dam with the Results up to the Age of 1 Year," *Proceedings*, Am. Concrete Inst., Vol. 30, pp. 485-497 (1934.).

By means of the suspension turbidimeter, developed at the Engineering Materials Laboratory of the University of California, it is possible to measure in one operation the specific surface of a ground material such as portland cement. The relative amount of light passing through a suspension of the material in a viscous liquid is measured by means of a photronic cell, a special type of photoelectric cell. The measurements may be made with sufficient accuracy to detect changes of 1 per cent in the specific surface of a given material. In this paper the application of the apparatus is confined strictly to portland cements, although studies are in progress using special cements and other granular materials.

There are at present ten sets of apparatus in use, covering both research work and routine plant control. Cooperative studies in progress will serve to perfect further the method and extend its application.

THEORY

The change in intensity of light upon passing through a liquid or suspension is herein called the turbidity. It can be shown that the change in turbidity due to the presence of suspended opaque particles in a suitable liquid is a measure of the surface area of the particles.

If the intensity of light transmitted through the clear medium be designated as I_o and the intensity of light transmitted through the suspension be designated as I_1, the change in the transmitted light, for opaque particles of equal size and very low concentration, is very nearly proportional to the concentration, or

$$I_o - I_1 = CM \text{ (approx.)} \qquad (1)$$

where M is the concentration or mass per unit volume and C is a proportionality constant. For small particles, such as those occurring in cements, even in low concentrations overlapping and shadowing of particles by other particles will occur and the change in the intensity of light will be logarithmic, as follows:

$$\log_e I_o - \log_e I_1 = CM \qquad (2)$$

or

$$\log_e \frac{I_o}{I_1} = CM \qquad (3)$$

For particles of given size, changing the concentration as expressed in Eq. 3 is equivalent to changing the surface area of particles in like percentage. If, however, the concentration and depth of suspension be held constant and the particle size be changed, the surface area of particles is again changed and the same law applies. Hence for a fixed concentration, there may be written an equation similar to Eq. 3, as follows:

$$C' \log_e \frac{I_o}{I_1} = KS \qquad (4)$$

where I_o is the intensity of light transmitted through the clear liquid, I_1 is the intensity of light transmitted through the suspension, C' is a factor dependent upon the concentration employed, S is the specific surface in square centimeters per gram, and K is the proportionality factor, called the *opacity factor*, which would be a constant if the particles were fully opaque. Under certain conditions and with certain modifications, the law applies to colored solutions as well as to suspensions of particles. The factor K defines the ability of the particles to absorb light and is influenced by the size and shape of particles.

It is assumed that the factor K is the same for all sizes of particles in the cement, or that the light absorbed is dependent only upon the projected area of the particles. Actually, the factor varies somewhat with the size of particles, due to their translucency. If the same factor is used for various finenesses of a given composition of cement, smaller differences than the differences in actual specific-surface values are obtained.

Since it is assumed that the opacity factor is the same for all sizes of particles, for a given cement the factor may be determined from a turbidity measurement on a sample of known size which has been retained between two sieves. It is necessary to evaluate the opacity factor only once for a given composition of cement, either by the method just described or by other methods to be described later.

With the opacity factor known, the test procedure is to determine the relative intensity of the light beam transmitted through the liquid containing a fixed concentration of the particles and the intensity

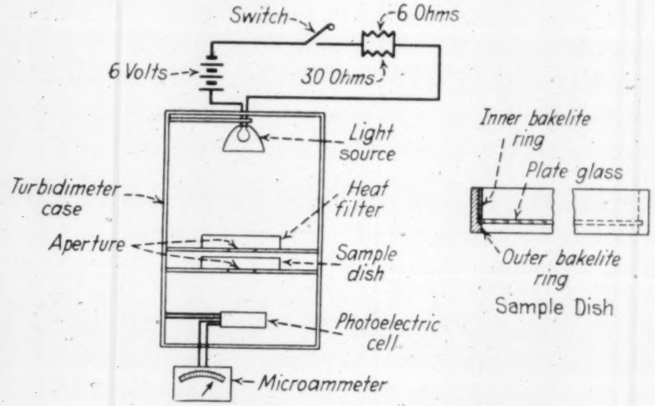
Switch
6 Ohms
6 Volts
30 Ohms
Light source
Turbidimeter case
Heat filter
Aperture
Sample dish
Photoelectric cell
Microammeter
Inner bakelite ring
Plate glass
Outer bakelite ring
Sample Dish

FIG. 1.—Diagram of Suspension Turbidimeter.

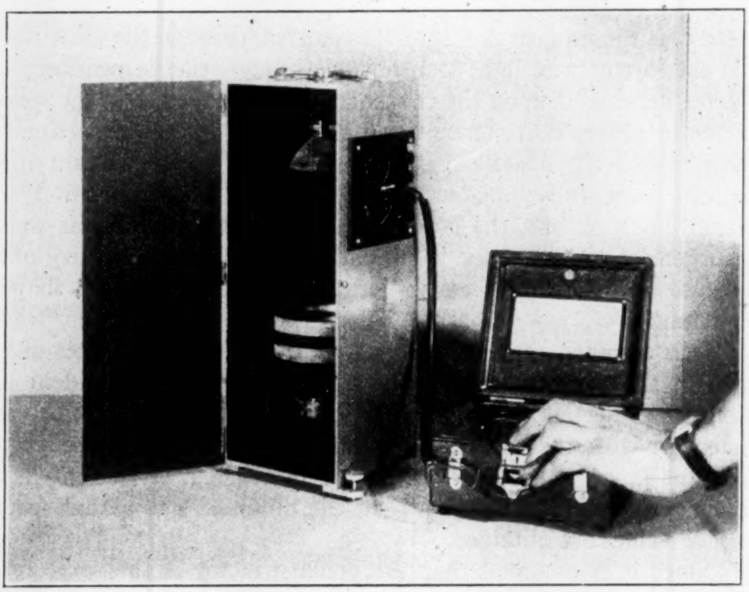

FIG. 2.—Suspension Turbidimeter.

of the light beam transmitted through an equal thickness of the clear liquid. The specific surface can then be calculated according to Eq. 4. Since the intensity of the light beam emerging from the clear liquid is a constant and need not be determined for each test, a single reading (on the suspension) is sufficient to complete the test.

The method of test does not depend upon any principle of sedimentation or upon the application of Stokes' law. Therefore, U.S.P. castor oil, which possesses the properties of high viscosity and natural dispersing power, is used as the suspending medium.

The basis for the preceding theory was summarized by Wells of the Bureau of Standards[1] who expressed the relationship between surface area and turbidity, and stated the principles involved in both suspension and sedimentation turbidimeters. In the use of his turbidimeter Wells calculated the rate of settling of silica particles out of suspension from the turbidimeter readings. Recently, L. A. Wagner[2] has further developed the use of a turbidimeter in determining particle-size distribution and surface area, employing principles similar to those evolved by Wells.

The turbidimeter described hereinafter, with respect to construction and operation, is similar to other turbidimeters. The distinguishing feature of this turbidimeter, as compared to others, lies in the method used to determine the proportionality factor K, without sedimentation.

APPARATUS

Figure 1 is a diagram of the apparatus showing all the essential parts required in construction. Figure 2 is a photograph of the assembled apparatus, ready for operation, together with the microammeter and sample dishes. A storage battery which furnishes current for the light globe is not shown. From a light source which is controlled by rheostats to constant intensity, a beam of parallel light rays is projected vertically downward through apertures. The beam, on passing through the apertures, impinges on a photronic cell which is connected to the terminals of a microammeter of low resistance. The sample, consisting of the suspension of cement in castor oil, is held in a glass-bottom container in the path of the light beam. The intensity of light passing through the suspension and affecting the sensitive cell is indicated by the microammeter. Between the light source and the sample is a heat filter consisting of a glass-bottom dish containing clear castor oil to a depth of approximately 4.1 mm. or 30.0 g. of oil in the container of $3\frac{7}{8}$ in. inside diameter. The bottoms of the sample dishes are all constructed from the same thickness of clear glass of good optical quality, and have the same light-transmission characteristics. All instruments are adjusted to give identical opacity factors for a given material, regardless of differences which exist in various photronic cells of the same type with respect to output and sensitivity; thus, nearly identical values of specific surface will be obtained with all instruments.

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[1] P. V. Wells, "Turbidity Standard of Water Analysis," National Bureau of Standards *Scientific Paper No. 367* (1920).

[2] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

PROCEDURE

The test procedure is, briefly, as follows: With only the heat filter in place, the intensity of light is so adjusted as to give a predetermined reading of the microammeter (50.0 microamperes, the maximum scale deflection). Cement in the amount of 0.250 g. is mixed in 50.0 g. of castor oil by agitating in a beaker, first by shaking the beaker and striking against the hand, and then by stirring with a spatula. All cement for test is passed through the No. 100 sieve, to facilitate dispersion. No chemical aid to dispersion is required. An amount of 25.00 g. of clear castor oil is weighed into a sample dish, the dish is placed in the path of the light beam, and a microammeter reading is taken. This dish is then replaced by a dish containing a 25.08-g.[1] sample of the cement and oil mixture, and a second microammeter reading is taken. The readings are made when the needle reaches a maximum and remains stationary, at which time the drift of the cell is complete and air bubbles induced by stirring have vanished. The two readings complete the test. The clear-oil reading need only be taken occasionally. After a test the sample dish containing the cement may be placed on the stage of a low-power microscope and the effectiveness of dispersion and absence of microscopic air bubbles may be checked.

CALCULATION OF SPECIFIC SURFACE

For the apparatus and method here described the specific surface is calculated by means of the equation:

$$S = \frac{1}{2K} \cdot \log_e \frac{I_o}{I_1} \qquad (5)$$

The solution of Eq. 5 involves the use of natural logarithms. However, common logarithms may be used by applying the conversion factor 2.303. A table or chart may readily be prepared, giving for any opacity factor the specific surface directly in terms of the microammeter readings.

Ordinarily the opacity factor is determined by a turbidimeter test on a fractionated sample, between the Nos. 200 and 325 sieves. Assuming the particles to be spherical and to have an average diameter d which is the arithmetic mean (0.00585 cm.) of the nominal sieve openings, the specific surface S in square centimeters per gram is given by:

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[1] The slightly higher weight of the dispersed sample is required to furnish a depth of suspension equal to the depth of 25.00 g. of clear oil, due to the differences in the specific gravities of oil and cement.

$$S = \frac{6}{pd} \quad (6)$$

where p is the specific gravity of the cement. Particles larger than the nominal sieve opening will pass a given sieve, but the particles are of irregular shape and the surface area per unit weight is approximately the same as for spheres of diameter equal to the nominal sieve opening. Thus, the errors arising from the assumptions as to size of particles in the fraction are largely compensating.

Particle-size distribution was found to have little effect upon the opacity factor for a particular composition of cement. There was found to be a close relation, however, between the specific gravity of the cement and the opacity factor. Based upon all the available fractionation factors, there was derived the following expression involving specific gravity alone:

$$K = (4.035\, p - 10.3)\, 10^{-4} \quad (7)$$

The data included cements ranging in specific gravity from 3.05 to 3.30 and ranging in color from white to very dark. Equation 7 is particularly useful where many cements are involved and where speed and economy in testing are important. Comparison of values of the opacity factor as determined by fractionation and as computed from the specific gravity shows an average difference of approximately 2 per cent.

Where there is available a cement of known specific surface as found by means of some other method of determination, the opacity factor may be evaluated by substitution of the known value in Eq. 5. In the Engineering Materials Laboratory, the specific surface of many compositions as determined by means of the microneter apparatus of the Riverside Cement Co. served to evaluate the opacity factors, in this case called microneter factors. The microneter factors for 55 laboratory cements of wide range in color and composition vary between 2.3×10^{-4} and 2.7×10^{-4}, assuming a specific gravity of 3.15 for all cements. The use of an average factor of 2.5×10^{-4} for all cements permits determinations of specific surface to be made with an extreme error of 8 per cent and a probable error of less than 3 per cent, without the necessity of determining individual factors. The extreme error may be considerably reduced, when the composition of the cement is known, by using average values of the factor equal to 2.2 × 10^{-4} for white cements, 2.4 × 10^{-4} for normal commercial cements, and 2.6 × 10^{-4} for commercial low-heat cements.

A comparison of the fractionation factor with the microneter factor shows an average difference of less than 3 per cent over the whole range of cements investigated, the average factors being practically identical as obtained by the two methods.

TABLE I.—REPRODUCIBILITY OF RESULTS.

Cement	Specific Surface, sq. cm. per g.[a]		Observer	Difference from Mean, per cent	
	Single Observation	Average		Observation	Average
L-21 (1300)	1290	....	No. 1	0.0	....
	1290	....	No. 2	0.0	....
	1290	....	No. 3	0.0	....
	1290	1290	No. 3	0.0	0.0
L-21 (1700)	1790	....	No. 1	0.2	....
	1790	....	No. 1	0.2	....
	1780	1786	No. 1	0.3	0.2
L-22 (1300)	1325	....	No. 2	0.6	....
	1350	....	No. 2	1.3	....
	1325	1333	No. 2	0.6	0.8
L-22 (1700)	1745	....	No. 2	0.9	....
	1760	....	No. 1	0.2	....
	1770	....	No. 2	0.9	....
	1750	1756	No. 4	0.3	0.6
L-23 (1600)	1605	....	No. 2	0.0	....
	1625	....	No. 2	1.3	....
	1580	....	No. 2	1.5	....
	1605	....	No. 1	0.0	....
	1605	1604	No. 3	0.0	0.6
L-24 (1300)	1340	....	No. 1	1.1	....
	1310	....	No. 2	1.1	....
	1340	....	No. 2	1.1	....
	1310	1330	No. 2	1.1	1.1
L-25 (1300)	1305	....	No. 1	0.4	....
	1290	....	No. 1	0.8	....
	1305	1300	No. 1	0.4	0.5
Average for all cements	....	....	....	....	0.54

[a] Specific surface calculated from fractionation factors.

COMPARISONS OF TEST RESULTS

Values of specific surface as determined by the suspension turbidimeter and by two sedimentation methods are presented and compared in Tables I to VI.

Tables I and II illustrate the degree of reproducibility of test results possible with the suspension turbidimeter. The cements indicated in Table I were ground in connection with the cement investigations for Boulder Dam. At the time these cements were ground, the suspension turbidimeter had not been fully developed, and control samples of given fineness were furnished by the Riverside

Cement Co. The microneter values for these control samples are shown in Table IV, under the heading of "Second Series." The values of specific surface shown in Table I were calculated by applying to the turbidity ratios obtained at that time, the fractionation factors obtained at a later date with the improved turbidimeter, and for that reason will not agree with the values in Table IV. As indicated in the table, turbidity ratios for the control samples were determined by four different observers with an extreme error of 1.5 per cent and an average error of 0.54 per cent.

Table II shows the effect, upon accuracy of determinations, of variation in the initial intensity of light, I_o. It is apparent that the initial intensity has little or no effect upon the accuracy of estimation of readings in the lower range of the scale. There appears to be slightly less difference in the upper third of the scale range than in the middle third, but the differences are not large. For the same reason as given in the previous paragraph, the value of specific surface of cement L-7 is not the same in Tables II and III.

TABLE II.—EFFECT OF INITIAL INTENSITY OF LIGHT IN REPRODUCIBILITY TESTS FOR CEMENT L-7.

INITIAL INTENSITY OF LIGHT, I_o, MICROAMPERES	SPECIFIC SURFACE,[a] SQ. CM. PER G.	DEVIATION FROM MEAN, PER CENT
50.0	1218	0.8
45.0	1218	0.8
40.0	1208	0.0
35.0	1228	1.0
30.0	1228	1.0
27.5	1218	0.8
25.0	1192	1.3
22.5	1200	0.7
20.0	1190	1.5
17.5	1222	1.2
Average	1208	0.9

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[a] Specific surface calculated from fractionation factor.

Referring to Tables III and IV, it is seen that there is excellent agreement between groups of determinations with regard to the relative finenesses of any given composition, and fair agreement with regard to the relative fineness of various cements. Approximately constant differences exist between the values of specific surface as determined by various methods. The summary of average values as related to the suspension turbidimeter, given in Table V, shows that the lowest relative values are obtained using the microneter and the suspension turbidimeter, which yield approximately equal results. The highest relative values are obtained using the Wagner turbidimeter of improved design, and the two sets of values for this instrument are approximately equal. Intermediate values are obtained using the Wagner turbidimeter of 1932 design, at the University of California.

TABLE III.—SPECIFIC SURFACE OF LABORATORY CEMENTS OF APPROXIMATELY EQUAL FINENESS, AS DETERMINED BY DIFFERENT METHODS.

Cement	Specific Surface, sq. cm. per g.				
	Suspension Turbidimeter	Microneter	Wagner Turbidimeter		
			University of California[a]	Bureau of Standards[b]	Bureau of Reclamation[b]
FIRST SERIES					
L-2	1145	1153	....	....	....
L-1	1245	1235	....	....	....
L-2a	1185	1183	....	....	....
L-3	1170	1194	....	....	....
L-3a	1220	1207	....	....	....
L-5	1150	1219	1261	1380	1435
L-6	1160	1185	1328	1400	1400
L-7	1160	1189	1325	1460	1455
L-9	1185	1225	1222	1470	1400
L-10	1180	1172	....	....	....
L-11	1125	1102	....	....	....
L-12	1155	1136	1248	1320	1275
L-14	1235	1208	....	....	....
L-15	1230	1202	....	....	....
L-16	1230	1209	....	....	....
L-19a	1090	1154	....	....	....
L-19b	1065	1139	....	....	....
Average	1172	1183	1277	1397	1389
HEAT-TREATMENT SERIES					
L-4-H[c]	1150	1147	....	....	....
L-4-RH[d]	1125	1164	....	....	....
L-4-2	1170	1126	....	....	....
L-13-H	1180	1173	....	....	....
L-13-RH	1150	1198	....	....	....
L-13-2	1185	1210	....	....	....
L-20-H	1145	1132	....	....	....
L-20-RH	1120	1130	....	....	....
L-20	1140	1173	....	....	....
Average	1152	1161	....	....	....

[a] Values obtained with Wagner turbidimeter of 1932 design.
[b] Values from U. S. Bureau of Reclamation *Technical Memorandum 371*, April 5, 1934.
[c] Symbol, H, denotes clinker heat-treated by continuous process.
[d] Symbol, RH, denotes reheated clinker.

Table VI illustrates the change in the opacity factor with particle size as measured by the equivalent sieve diameter. Where the average size of particles below 8 μ is calculated using the fractionation factor obtained from test on particles retained between the Nos. 200 and 325 sieves, the resulting average equivalent sieve diameter,

TABLE IV.—SPECIFIC SURFACE OF LABORATORY CEMENTS OF VARYING FINENESS, AS DETERMINED BY DIFFERENT METHODS.

Cement	Specific Surface, sq. cm. per g.				
	Suspension Turbidimeter	Microneter	Wagner Turbidimeter		
			University of California[a]	Bureau of Standards[b]	Bureau of Reclamation[b]
FINENESS SERIES					
L-4 (1000)	1005	970	1073	1200	1060
L-4-2 (1200)	1170	1126	1360	1480	1305
L-4 (1400)	1375	1331	1486	1580	1640
L-4 (1600)	1585	1537	1808	1910	1780
L-13 (1000)	980	980	1042	1130	1075
L-13-2 (1200)	1185	1210	1349	1450	1360
L-13 (1400)	1365	1368	1522	1630	1630
L-13 (1600)	1600	1548	1719	1840	1840
L-20 (1000)	930	918	984	1140	1130
L-20 (1200)	1140	1173	1295	1350	1370
L-20 (1400)	1360	1398	1603	1640	1665
L-20 (1600)	1520	1527	1762	1830	1920
Average	1268	1257	1417	1515	1481
SECOND SERIES					
L-21 (1300)	1265	1300	....	....	1520
L-21 (1700)	1650	1699	....	....	2080
L-22 (1300)	1295	1309	....	....	1545
L-22 (1700)	1690	1712	....	....	2035
L-23 (1600)	1665	1607	....	....	1890
L-24 (1300)	1250	1302	....	....	1480
L-25 (1300)	1260	1309	....	....	1535
L-25 (1360)	1320	1355	....	....	1580
Average	1424	1449	....	....	1708

[a] Values obtained with Wagner turbidimeter of 1932 design.
[b] Values from *Technical Memorandum 371*, U. S. Bureau of Reclamation, April 5, 1934.

TABLE V.—COMPARISON OF TEST RESULTS BASED UPON AVERAGE VALUES FOR CEMENTS IN TABLES III AND IV.

Series	Percentage of Suspension—Turbidimeter Value			
	Microneter	Wagner Turbidimeter		
		University of California	Bureau of Standards	Bureau of Reclamation
First series	101.0	110.5	121.0	120.1
Heat-treatment series	100.9	.....	.....	.....
Fineness series	99.2	111.9	119.6	117.0
Second series	101.6	.....	.....	119.8
Weighted average	100.6	111.2	119.7	118.3

TABLE VI.—EFFECT OF PARTICLE SIZE UPON OPACITY FACTOR.

Range of Particle Size, μ	Average Particle Size, μ	(Assuming $K = 2.47$) Computed Particle Size, μ	(Assuming Average Particle Size) Computed Opacity Factor $K \times 10^4$
210-147	178.5	121.5	3.62
147-104	125.5	91.0	3.40
104-74	89.0	72.0	3.04
74-43	58.5	58.5	2.47
Below 8	.....	5.3	....

5.3 μ, agrees with that assumed in the microneter method (5.35 μ), although there is evidence from other sources that the actual average size of these particles is considerably lower. This fact accounts for the close agreement between the average microneter and average fractionation factors, where the microneter calculations are made in accordance with the customary assumptions. The fraction below 8 μ was obtained by sedimentation in absolute alcohol. The settling time for particles coarser than this fraction was computed from Stokes' law, the time being determined by comparison with the time required for the fall of particles having a minimum equivalent sieve diameter of 43 μ. The suspension containing the fine fraction was drawn off after the time computed for particles coarser than 8 μ to pass a given depth, and this suspension was then evaporated to obtain the sample.

CONCLUSIONS

Through the use of the suspension turbidimeter, results are obtained which furnish adequate verification of the theory upon which this method of determining specific surface is based. The apparatus possesses the following advantages:

1. The instrument is economical, with respect both to initial cost and to the cost of operation and maintenance.
2. The degree of reproducibility of test results is adequate for all practical purposes. Results within 1 or 2 per cent are obtained by various observers.
3. A high order of skill is not required for the operation of the apparatus.
4. Determinations of specific surface are rapidly made, a complete test requiring only 20 min. Calculations are simple and direct, and may be arranged in the form of a single chart.
5. Neither sieve analysis nor standard samples are required.
6. Individual instruments can be so adjusted as to produce practically equal results on the same sample of cement.
7. The operation of the apparatus is little affected by the properties of the dispersing medium and is independent of temperature. The time element does not enter into the measurements.
8. The instrument is portable and is particularly adaptable to commercial plant control and routine testing.

The method possesses the disadvantage that particle-size distribution cannot be determined. However, it is believed that the specific surface is an adequate measure of the fineness of cement for most practical purposes.

DISCUSSION

Mr. L. A. Wagner.[1]—Standing in the way of all turbidimeter-particle size work is the necessity of evaluating a property of each cement which I called its "transmittancy constant" and which the present author calls its "opacity factor." The present author has developed three methods for evaluating this constant.

The first consists in separating a fraction of the sample between the No. 200 and No. 325 sieves and determining a factor for this fraction. In Table VI of his paper he shows that reducing particle diameter three fold causes a reduction in opacity factor of 30 per cent. In spite of this rapid change of factor with particle size, he assumes that a factor obtained from 60 μ particles is valid for particles 10 or 20 diameters smaller. The validity of this assumption is doubtful.

The second method is a correlation which the author has established between the opacity and density of cement. It is well known that the color of a cement is greatly changed by small variations in the method of cooling of the clinker. Hence it may often occur that cements of identical densities will have widely differing colors and opacity factors. I believe this method for determining the factor is also of doubtful value.

The third method consists in first making an analysis of the cement in the microneter and from the specific surface thus obtained a factor may be calculated which when applied to turbidimeter data for the same cement will give the same answer. With this method I find no criticism. The microneter is one of the most precise instruments ever developed for particle size determination and any factor obtained from it should be dependable.

In conclusion it is well to emphasize again that at present we have no method for determining an *absolute* value of the surface area of particles as small in size and irregular in shape as those of portland cement. With the microneter as well as with the turbidimeters the average diameter of the smallest particles is arrived at only by assumption.

Mr. P. S. Roller.[2]—The size fraction between the No. 200 and No. 325 sieve is much darker in color than the finer fractions of cement. Therefore, it is obviously a serious error to attempt to calibrate the apparatus with this fraction without correcting for color.

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[1] Assistant Chemical Engineer, Cement Reference Laboratory, National Bureau of Standards, Washington, D. C.

[2] Associate Chemist, Nonmetallic Minerals Experiment Station, U. S. Bureau of Mines, New Brunswick, N. J.

It is estimated by the author that even though "particles larger than the nominal sieve opening will pass a given sieve" nevertheless the surface area is the same as that of an equivalent sphere of diameter equal to the opening. To say that the particles are of irregular shape is a peculiar reason to give for this assumption, since the more irregular the particle the greater the surface area per unit volume. The assumption is readily disproved by actual measurement.

In attempting to translate measurement of light scattering specifically to particle size, two questions must be considered carefully, namely, whether the scattering is linearly proportional to the surface concentration and whether deflocculation is complete. I do not believe that the first question can be answered in the affirmative. Lowry and Marsh[1] have carefully measured the light transmission of sized fractions of ground barites suspended in medicinal paraffin. They concluded that for the same fraction the optical density is directly proportional to the surface concentration. For different fractions the "optical density is proportional to surface concentration of the powder but increases slowly as the diameter is reduced to 50 μ and much more rapidly when the diameter is reduced below 30 μ."

There is probably some merit in using castor oil as a suspension medium in view of a probable natural deflocculating action. However, definite assurance of this should be given, especially with respect to the effect of stirring, absorbed moisture, rancidity, and possible reaction with the high-lime cement grains. It may be mentioned as of interest in this connection that the deflocculating action of oleic acid (in benzene) is upset completely by traces of dissolved moisture.[2]

The comments on translation of turbidimetric measurements specifically into particle size are of import both in general and for cement technology. In view of the results of Lowry and Marsh and other similar results, I believe that measurement of optical opacity as heretofore carried out with reference to portland cement gives values for fineness that are much too high, unless there is accidental compensation due to partial flocculation or other cause.

MR. R. W. CARLSON.[3]—The unique feature in the determination of fineness by means of the suspension turbidimeter is the sieve-fractionation method of evaluating the opacity factor. It happens that if the particles passing a No. 200 sieve and retained on a No. 325

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[1] Lowry and Marsh, *Transactions*, Faraday Soc., Vol. 24, p. 195 (1928).

[2] Harkins, Gans, and Ryan, *Industrial and Engineering Chemistry*, Vol. 24, p. 1288 (1932).

[3] Research Engineer, Engineering Materials Laboratory, University of California, Berkeley, Calif.

sieve are assumed to have the same specific surface as spheres of diameter equal to the average sieve opening of these two sieves, the suspension turbidimeter gives results which agree with the microneter. But there is another rather accidental fact which helps to bring about the agreement. In the microneter method all particles finer than 8 μ are assumed to have a diameter of 5.35 μ in the calculations. These particles probably have an average diameter of less than 5 μ. Therefore the specific surface values as ordinarily computed from microneter data may be low, and the finer the cement, the lower the values are in comparison to actual values. The suspension turbidimeter gives similar results but for a different reason. The finer particles are less opaque than the larger particles but since the same opacity factor is used for all finenesses of cement the fine cements are not shown to be sufficiently fine. Thus it is seen that through choosing a particular assumption in determining the opacity factor, in combination with the fact that both microneter method and the suspension turbidimeter method do not give full weight to the very fine particles, the microneter and suspension turbidimeter yield comparable results.

MR. J. R. DWYER.[1]—In Table III of Mr. Klein's paper it is shown that the microneter and the suspension turbidimeter results agree very closely. Apparently the difference between them is in the order of one to three per cent of the suspension turbidimeter values. In this year's report[2] of the fineness subcommittee of the Society's Committee C-1 on Cement are the results of four samples used in comparative sub-sieve fineness tests, and there the differences between the microneter and the suspension turbidimeter vary from about 100 to practically 200 sq. cm., that is, from about 6 to 13 per cent. I was wondering what might be the reason for this difference in relative performance.

MR. P. H. BATES.[3]—Assumptions were made in this paper with regard to microneter and turbidimeter as to the sizes of the various fractions. They might apply to the data presented in the paper, but in these data the microneter values are low and the turbidimeter values are high, whereas in the data in the report of Committee C-1 it is the reverse. I want to call particular attention to that, because in some data the Bureau of Reclamation has presented they have given factors for the conversion of microneter values to turbidimeter values, but those factors will not apply at all according to the data of Committee C-1.

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[1] Research Associate, National Bureau of Standards, Washington, D. C.

[2] Report on Cooperative Sub-Sieve Fineness Tests by the Working Committee on Fineness, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 315 (1934).

[3] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

The Bureau of Reclamation dry all their samples, and they maintain that when they dry a sample and then use it in the turbidimeter, they can reproduce their results month after month.

MR. BRADLEY DEWEY.[1]—In connection with some research, we are using three Wagner instruments and three of the Klein instruments, one of each being used in our home laboratory and two by widely separated field men in plants. We are enthusiastic believers in the Wagner instrument, its usefulness, and its value in connection with the study of particle size distribution. However, where a mill wishes to control grinding to fairly narrow limits of particle size, the Klein instrument has great value. It is very easily used, it can be turned over to a laboratory boy, who can be taught how to use it in a very short time and obtain results that, for many purposes, are all that are wanted. The reproducibility between results has been very close in different laboratories, though our work has been going along at high speed without any particular care.

There are, however, some mysteries, too. One of them is that some of our samples check very closely with the Wagner and some do not. In some cases our Wagner readings run as much as 200 higher, whereas in others we have almost a perfect check. For example, with the last Bureau of Standards sample that was, if I remember correctly, somewhere in the 1400 range, we obtained practically perfect checks right through the range on three Klein instruments and two Wagner instruments. On the other hand, we have not been able to do that with all our samples. But where a mill is using it to control their own fineness, it does not make very much difference to them what the value of K is. Where it is being used for research work, that is an all-important feature and is something that will have to be ironed out. But I would recommend that the instrument be given serious consideration where volume of output in the hands of fairly cheap help is a factor. Turbidimeters are not necessarily precision instruments for research, but should be considered as substitutes for the No. 200 sieve.

MR. H. R. BRANDENBURG[2] (*by letter*).—One of the first units of apparatus constructed of the design described in the paper has been in use by the Cowell Portland Cement Co. for about a year.

As stated in the paper it is required that a "fraction" be obtained perfectly free from flour so that a correct opacity factor may be obtained. Depending upon the history of the cement this operation may be more difficult with some than with others; but as a common requirement cements must be shaken to refusal on the No. 325 sieve and the fraction thereafter rolled in glass to eliminate all remaining traces of flour. When thus properly prepared it is possible to obtain repeatedly concordant results with freshly prepared fractions of the same cement.

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[1] President, Dewey & Almy Chemical Co., Cambridge, Mass.

[2] Chief Chemist, Cowell Portland Cement Co., Cowell, Calif.

It has been definitely determined that the apparatus, using the described method of procedure, gives consistently reproducible values. An easily acquired manipulative expertness and careful work will not only result in the ability to readily reproduce results but will also prove the degree of precision of which the instrument is capable.

The following *K* and specific surface results have been taken at random from determinations made on various cements, domestic and foreign, and may serve to illustrate the reproducibility obtained in our laboratory:

Cement	Values for Factor *K*		Specific Surface, sq. cm. per g.	
	Original	Check Values	Original	Check Values
A	2.56	2.55	1164	1169
B	2.58	2.56	1277	1283
C	2.72	2.72	1355	1350
D	2.72	2.68	1175	1195
E	2.44	2.48	1173	1158
F	2.70	2.68	1150	1148

From certain considerations we would also say that the opacity factor *K* seems to represent a value which appears to have other than a mere mathematical meaning. For, although fractions of the same cement were checked and rechecked within the limits of the "possible" instrumental error it was found that *K* varied between different lots from the same manufacturer. These variations, while not large, were proved to exist and were suggestive of some variation, the cause of which conceivably resided in the physical or chemical nature of the product, even though the potential compound composition remained essentially the same.

From experimental work it has been concluded that *K* expresses a value which may match in usefulness its initially intended function as a constant in the specific surface equation. It appears that *K* on the same clinker can be artificially caused to change its value by one means or another. Extreme changes in *K* have been brought about, for instance, by taking the clinker upon its discharge from a kiln and allowing one part to air cool slowly while rapidly chilling the other portion in an excess of water, the values of *K* varying in their extremes from 2.82 for the air-cooled down to 2.24 for the chilled product. Operating conditions in a kiln varying from normal, whether caused intentionally or otherwise, may cause similar divergences from the normal *K* value, although not of so pronounced a nature as those indicated above.

Further investigations, it is believed, will establish the fact that a maximum *K* value is assignable to any given compound composition, provided the clinker has been manufactured under optimum burning and normal cooling conditions. From the same potential compound composition it is possible by means of variations of control during the burning stage, the cooling stage and any heat treatment to produce clinkers varying in color, mineralogical structure and physical characteristics, which variables are reflected in changes of the opacity factor.

MR. ALEXANDER KLEIN[1] (*author's closure by letter*).—The preceding discussion, while not in disagreement with the basic idea that the relative light transmission furnishes a measure of fineness, concerns itself chiefly with the validity of the specific assumptions and with the correlation of test results as obtained by means of different instruments. The fact remains that over a wide range of cements the suspension-turbidimeter results are consistently in excellent agreement with those obtained by microneter, which instrument is stated by Mr. Wagner to be "one of the most precise instruments ever developed for particle-size determination."

The author cannot concur entirely with Mr. Wagner's statement that "it may often occur that cements of identical densities will have widely differing colors and opacity factors." This is *rarely* the case, appreciable changes in density following only upon changes in composition of clinker or abnormal increases in ignition loss. Table III shows results for cements made from heat-treated clinkers in which the color changed upon heat treatment from dark iron-gray to light tan or straw color; the extreme change in specific gravity was 0.03 and there was no apparent change in the opacity factors other than that corresponding to the change in specific gravity. If conditions of clinker treatment are such as to change markedly the value of the opacity factor, as reported by Mr. Brandenburg, there are marked changes in composition, density, or both. For the 55 cements reported herein, ranging from very light to very dark, the extreme difference between the density factor and the microneter factor is 4 per cent and the average difference is 2 per cent.

Mr. Roller's suggestion that a correction for color be made in evaluating the fractionation factor does not apply. The fractions differ not in color but in shade only, and the absorption of light by the particles of various sizes differs not qualitatively but quantitatively.

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Messrs. Wagner and Roller refer to the variation in light transmission for different size fractions, as casting doubt upon the propriety of using a single opacity factor for a given material. However, for a given total surface area a decrease in particle size is accompanied by an increase in the number of particles, and within the proposed range of use the *total* light absorbed tends to remain substantially constant. A confirmation of the stability of the opacity factor is furnished by the results reported in Table IV, fineness series. In this series both the fractionation and micronéter factors remain uniform although the specific surface and particle-size distribution vary considerably. Further, for the 55 cements covered by this report, the extreme difference between the fractionation factor and the micronéter factor is 3 per cent.

Mr. Bates and Mr. Dwyer point to a difference between the relative results obtained by suspension turbidimeter and micronéter as reported herein and as reported for four cements by Committee C-1. In calculating the values of fineness by micronéter for the report of Committee C-1, the assumptions were not those usually employed for the micronéter but were those designated by the subcommittee on fineness. Employing the usual assumptions for micronéter calculations, Mr. Hubert Woods of the Riverside Cement Co. has calculated the specific surface of the C-1 cements to be as shown in the tabulation below. The C-1 values obtained by suspension turbidimeter are shown for comparison:

	SPECIFIC SURFACE, SQ. CM. PER G.			
	No. 23678	No. 23679	No. 23680	No. 23934
Micronéter	1450	1605	1813	1620
Suspension turbidimeter	1455	1625	1820	1640

The agreement is close. The difference in relative results as between the present paper and the C-1 report lies not in instrument performance but in the assumptions for calculation.

Since all agree that the absolute value of specific surface cannot be determined by measurement, there is little point to discussion regarding the *actual* surface areas of fine irregular particles.

Mr. Roller questions the use of castor oil with respect to effect of stirring, absorbed moisture, rancidity of oil, and possible reaction between oil and suspended material. These possible effects were considered during the development of the apparatus, and under the usual procedure of testing they either are eliminated or are of negligible amount.

It is gratifying to note that Messrs. Dewey and Brandenburg have found the suspension turbidimeter to be useful in actual service.

THE VANE CALORIMETER

By R. W. Carlson[1]

Synopsis

This paper describes a type of calorimeter which can be used for measuring heat of hydration of neat-cement pastes while curing at substantially constant temperature. This calorimeter has certain advantages over other types of calorimeters, chief among which are its low cost and its suitability for cements which are not readily soluble in acids.

Typical data are presented with diagrams showing the hourly rates of heat evolution for a number of cements plotted against the time after mixing. Comparison of results from this calorimeter and from the heat-of-solution calorimeter indicate that satisfactory accuracy is obtained using the vane calorimeter.

Introduction

The vane calorimeter is an apparatus for determining *rates* of heat liberation during the hardening of neat-cement paste. From these rates the total amount of heat liberated up to any age is computed. Precise measurements are made, at intervals, of the difference in temperature between the specimen and a surrounding surface, which surface is maintained at a constant temperature and is separated from the specimen by metal vanes.

The method of determination differs from others in that measurements are made on a fairly large neat-cement specimen, cured at a substantially constant temperature. The differences in temperature between specimen and constant-temperature surface are sufficiently small that they do not appreciably affect the rate of hydration, but are sufficiently large that they can be accurately measured by means of resistance thermometers. The method is suitable for cements which cannot be dissolved in acid, as required in the heat-of-solution method. It is, therefore, adaptable for cements other than portland, such as pozzuolanic cements.

Description

The vane calorimeter proper consists of four essential parts, which are shown in Fig. 1: (1) a central metal cup in which the specimen (in a sealed metal can) is placed, (2) metal vanes which radiate

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from the cup to (3) a surrounding copper shell, and (4) resistance thermometers intimately secured to the cup and to the shell. An insulating material, such as kapok, fills the space between the vanes. A bakelite chimney permits specimens to be dropped into the cup even when the calorimeter itself is buried; and in this chimney an insulating plug is inserted when the specimen is in place.

In making a test, a sealed can of neat-cement paste (usually 800 g. of cement) is placed in the metal cup. As the specimen liberates heat, it rises in temperature slightly and the heat is conducted through the metal vanes to the copper shell. The difference in temperature between the cup and the shell is proportional to the rate at which heat is being conducted away from the specimen. This difference is determined from a single observation using a special test set. The test set operates on the Wheatstone bridge principle and measures the *ratio* of resistances of thermometers on the cup and similar thermometers on the shell. Also, the temperature of the specimen is determined from the thermometers on the cup. The thermometers are in duplicate, one on each side of the cup (180 deg. apart) and one on each side of the shell, and are connected in series; this arrangement averages the temperatures on opposite sides of the specimen. The thermometers are made of copper wire wound on copper or brass spools and are thoroughly aged before installation.

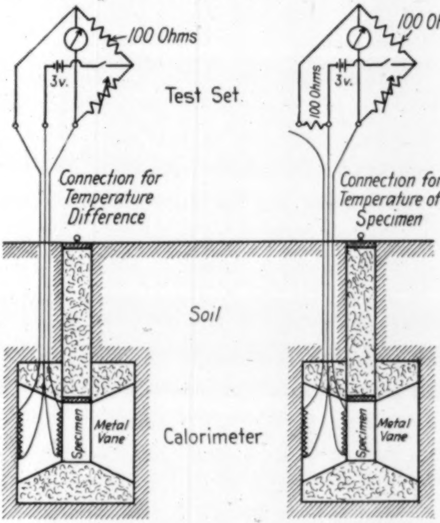

100 Ohms
3v.
Test Set
100 Ohms
3v.
100 Ohms
Connection for Temperature Difference
Connection for Temperature of Specimen
Soil
Specimen
Metal Vane
Calorimeter
Specimen
Metal Vane

FIG. 1.—Vane Calorimeter.

In order to calibrate the calorimeter, a resistance unit is placed in the metal cup and is connected to a storage battery. Electrical measurements are made to determine the rate of heat output by the resistance unit and, simultaneously, the temperature difference which develops between the metal cup and the copper shell is measured in terms of the resistance ratio. When the temperature difference reaches an equilibrium value, which may require a few hours, the heat is being conducted away from the metal cup at the same rate as it is being released from the resistance unit. The rate of heat removal per degree of temperature difference is then computed.

One step in the computations can be eliminated by not computing the temperature difference from the measured resistance ratio; in such a case the rate of heat removal which results in a 1-per-cent change in resistance ratio is computed and called the "calibration constant." Another simplification can be made if the same amount of cement is used in each test. The calibration constant is then computed in terms of the heat-removal rate, per gram of cement, which causes a change in resistance ratio of 1 per cent.

Due to the heat capacity of the specimen (and the metal cup), heat is not conducted from the specimen at the same rate as it is liberated by the hydrating cement. When the rate of heat liberation is increasing, some heat is being stored in the specimen. The rate of this heat storage is equal to the rate of temperature rise multiplied by the heat capacity per gram of cement. It could be entirely neglected without causing serious error in the heat of hydration except during the first few hours, since the heat storage over a period of time is negligible.

The maximum rate of heat liberation of portland cement may be more than one hundred times the minimum rate. In the tests to date, the maximum observed rate is 14 cal. per g. per hr. After the age of 28 days, the rate may be only a few hundredths of a calorie per gram per hour. Therefore, it is advisable to have one calorimeter with heavy vanes for early periods when the rate is high, and another more sensitive calorimeter with lighter vanes for the later periods.

Detailed Procedure and Computations:

Somewhat more than 800 g. of cement are mixed with 40 per cent, by weight, of water, all at the temperature of the calorimeter. Exactly 1120 g. of the paste are then placed in a 3 by 6-in. metal can and sealed. This specimen is placed in the calorimeter and the chimney plug is inserted. Readings are begun when the specimen is $1\frac{1}{2}$ hr. old, and are repeated each hour until a maximum rate of heat liberation is reached. Thereafter the readings are taken only often enough to permit a curve to be drawn of the relation between rate of heat liberation and age.

Having obtained the readings for the specimen, the rate of heat removal is computed from the resistance ratio, and the rate of heat storage is computed from the temperature change of the specimen. The sum of these rates, in calories per gram per hour, is the actual amount of heat liberated by a gram of the cement in one hour. The total amount of heat liberated at any age is then the sum of the hourly rates up to that age. This is called the "heat of hydration" of the cement. The principal importance of the heat of hydration lies in its direct relation to the rise in temperature of hardening concrete.

TABLE I.—SAMPLE COMPUTATION SHEET FOR VANE CALORIMETER.

NOTES.—Calorimeter calibration constant is 1.77 cal. per g. per 1 per cent change in ratio. Heat capacity is 0.40 cal. per g. per 1 F. (0.72 cal. per g. per 1 C.).

Hours after Casting	Change in Resistance Ratio, per cent	Rate of Heat Removal, cal. per g. per hr.	Temperature of Specimen	Change in Temperature	Rate of Heat Storage, cal. per g. per hr.	Total Rate of Heat Liberation, cal. per g. per hr.	Heat of Hydration, cal. per g.
0.0	.....	....	69.0 F. (20.6 C.)[a]	....	....	....	2.00[b]
0.5	0.200[a]	0.36	....	0.0	0.00	0.36	....
1.0	.....	....	69.0 F. (20.6 C.)[a]	....	....	....	2.36
1.5	0.290	0.51	....	0.0	0.00	0.51	....
2.0	.....	....	69.0 F. (20.6 C.)	....	....	....	2.87
2.5	0.385	0.68	....	0.0	0.00	0.68	....
3.0	.....	....	69.0 F. (20.6 C.)	....	....	....	3.55
3.5	0.510	0.90	....	0.2 F. (0.1 C.)	0.08	0.98	....
4.0	.....	....	69.2 F. (20.7 C.)	....	....	....	4.53
4.5	0.635	1.12	....	0.8 F. (0.4 C.)	0.32	1.44	....
5.0	.....	....	70.0 F. (21.1 C.)	....	....	....	5.97
5.5	0.785	1.39	....	1.0 F. (0.6 C.)	0.40	1.79	....
6.0	.....	....	71.0 F. (21.7 C.)	....	....	....	7.76
6.5	0.945	1.67	....	1.0 F. (0.6 C.)	0.40	2.07	....
7.0	.....	....	72.0 F. (22.2 C.)	....	....	....	9.83
7.5	1.045	1.85	....	0.6 F. (0.3 C.)	0.24	2.09	....
8.0	.....	....	72.6 F. (22.6 C.)	....	....	....	11.92
8.5	1.090	1.93	....	0.4 F. (0.2 C.)	0.16	2.09	....
9.0	.....	....	73.0 F. (22.8 C.)	....	....	....	14.01
9.5	1.080	1.91	....	−0.1 F. (−0.05 C.)	−0.04	1.87	....
10.0	.....	....	72.9 F. (22.7 C.)	....	....	....	15.88

[a] Extrapolated value.

[b] Immediate heat of hydration measured by separate test.

The computations to obtain heat of hydration may be made clear from the sample form shown in Table I. In the first column are recorded the hours after casting the specimen. The values in the second column are obtained by subtracting from the observed resistance ratios the ratio corresponding to no heat removal or, in other words, no specimen in the calorimeter. These values are then multiplied by the calibration constant to obtain the rates of heat removal in the third column. The observed temperatures of the specimen are shown in the fourth column and the computed changes during each hour in the fifth column. The latter are multiplied by the heat capacity per gram of cement per degree Fahrenheit to obtain the rates of heat storage in the sixth column. The rates of heat removal and of heat storage are added to give the total rate of heat liberation in the seventh column. The rates are useful in comparing the hydration characteristics of cements, but more often the total amounts of heat liberated up to various ages are desired. These amounts are obtained by adding individual rates cumulatively from the beginning, and are recorded in the last column. The value at the top of the last column is not zero because the cement liberates an appreciable amount of heat immediately upon being mixed with water. This amount is calculated from the rise in temperature which is observed when water is added to the amount.

In order to standardize the calculations, the temperatures and the rates of heat removal (computed from the resistance-ratio readings) are plotted. This graphical method permits values at even time intervals to be determined and entered on a form such as that shown in Table I, even when only occasional readings are taken.

Control Required:

As indicated in Table I, measurements of resistance ratio are made to the nearest 0.005 per cent. This precision is not difficult to attain, since the conditions are constant and the two resistances entering into the measurement are nearly equal. However, this least reading corresponds to a temperature difference of only about 0.02 F. (0.01 C.) Therefore, accurate results cannot be obtained if there are fairly rapid variations in the temperature of the copper shell of more than 0.02 F. (0.01 C.) For curing at 100 F. (38 C.), it has been found satisfactory to house the calorimeters in a room thermostatically controlled within one or two degrees Fahrenheit (1 C.), if several inches of insulating material of high heat capacity are placed around the calorimeter. Even this insulating material is only sufficient when the variations in room temperature are more or less periodic so that they can be damped out. Better results have been obtained when the room has been controlled to about 0.1 F. (0.05 C.) and the insulating material around the copper shell retained.

For lower curing temperatures, burying the calorimeter in the ground beneath the floor of a room has been found to be most satisfactory. At a depth of 18 in. below the floor of a room controlled approximately to 70 F. (21 C.), variations of only 0.02 F. (0.01 C.) have been observed throughout a typical day. Even below the floor of an uncontrolled room, the temperature changes very slowly, and satisfactory results can be obtained. Installation underground appears to be the most economical and satisfactory arrangement. It is, of course, necessary that the heat liberated by cement at the lower curing temperature be related to that liberated by the same cement under the temperature conditions of use, as in mass concrete.

TYPICAL TEST RESULTS

A comparison of the heats of hydration observed for two curing temperatures, 100 and 70 F. (38 and 21 C.), is sh[illegible] two cements of different composition in Fig. 2. Both cements are normal except that the lime content of one is above normal and that of the other is below normal. It is at once apparent that the heat of hydration for either cement is lower for the lower curing temperature. At the age of 28 days, however, the difference between curing at 70 F. (21 C.) as compared with curing at 100 F. (38 C.) is negligible for the high-lime cement and not large for the low-lime cement. It is found from other tests that while the condition of curing constantly at 100 F. (38 C.) gives approximately the same heat of hydration as does curing at the variable temperatures of mass concrete for low-heat cements, this curing condition gives low results for other cements. For all types of cement, it is found that by adding 10 cal. per g. to the heats of hydration obtained at a curing temperature of 70 F. (21 C.), these values are more nearly equal to the heat of hydration for mass-concrete temperatures than are the values obtained at a curing temperature of

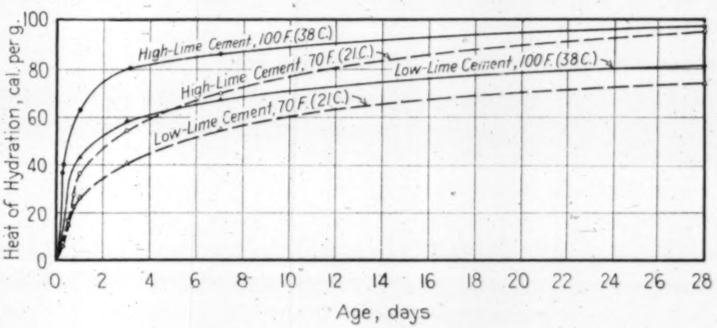
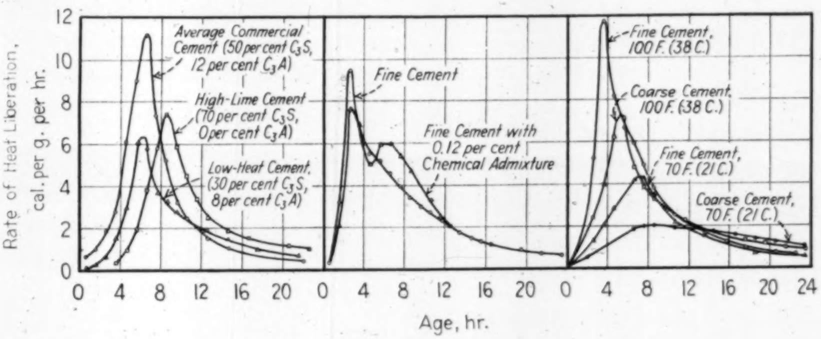


FIG. 2.—Heat of Hydration of Two Cements at Two Control Temperatures.



(a) (b) (c)

FIG. 3.—Typical Variations in Rates of Hydration.

100 F. (38 C.). It thus appears that the heat of hydration as determined from tests in a buried calorimeter (approximately 70 F., 21 C.) may be used as an indication of the probable heat of hydration in mass concrete.

For many studies, the *rates* of heat liberation, because of their greater sensitivity to influences such as composition of cement, are more useful than are the cumulative heats of hydration. In Fig. 3 are given examples showing the effects of various factors upon the rates of heat liberation during the first day of cement hydration. Figure 3 (*a*) shows the variations in rate which may be expected from variations in chemical composition of the cement when hydration is at 100 F. (38 C.). Figure 3 (*b*) shows the effect of an accelerating admixture upon the rates of heat liberation of an extremely fine cement, again at 100 F. (38 C.), and Fig. 3 (*c*) shows the effect of fineness upon the rates, both for curing at 70 F. (21 C.) and for curing at 100 F. (38 C.).

The reliability of results from the vane calorimeter has been ascertained by a limited number of comparative tests with the heat-of-solution calorimeter on specimens stored continuously at 100 F. (38 C.). At ages of 3 to 28 days, the maximum observed difference has been 3 cal. per g. or about 4 per cent. This agreement is satisfactory for most purposes.

The vane calorimeter possesses the disadvantage that, as a specimen continues to hydrate and its rate of heat liberation diminishes, the measurement of this rate becomes less and less accurate. For the most sensitive calorimeter thus far constructed, the least reading of resistance ratio which can be considered reliable corresponds to a rate of heat removal of 0.0025 cal. per g. per hr., or about 0.06 cal. per g. per day. Since this may be about one fourth the rate of hydration at the age of 28 days for an average cement, the error in rate measurement at this age may be about 12½ per cent. Although this error amounts to only about 1 cal. per g. in the heat of hydration up to the age of 28 days, it accumulates to 6 cal. per g. at the age of 6 months. Thus the calorimeter as ordinarily constructed is limited to tests up to the age of perhaps 28 days.

SUMMARY

The vane calorimeter has been developed to meet the need for an apparatus for measuring both the rate and the total amount of heat liberation of cements under a variety of conditions. Its construction cost is low and its operation is simple. Pozzuolanic cements may be tested as well as portland cements. Results are obtained with sufficient accuracy for most purposes, up to the age of 28 days and perhaps greater.

EFFECT OF DURATION OF MOIST CURING ON THE PRINCIPAL PROPERTIES OF CONCRETE

By A. G. Timms[1]

Introduction

The literature on concrete contains a wealth of information on the effect of duration of moist curing on strength, but little information relating to its effect on such properties as durability and volume change. This investigation was undertaken to supply some of the needed information.

The data cover strength tests at from 1 day to 2 years of 6 by 12-in. concrete cylinders moist cured the entire time and part of the time; strength tests of 6-in. concrete cubes after different numbers of cycles of freezing and thawing; loss in weight of 6-in. concrete cubes subjected to freezing and thawing; tests of permeability by capillary flow; and length change and absorption tests on 3 by 3 by 15-in. mortar bars. The tests were conducted with a normal portland cement and two high-early-strength portland cements.

Materials and Methods

The normal cement consisted of a mixture of equal parts of four brands of portland cement purchased in Chicago. Results for high-early-strength cement A are based on tests of three high-early-strength cements, two of the same brand manufactured at different plants, and the third a high-early-strength cement of similar strength characteristics. Separate tests on these three cements showed very satisfactory agreement. High-early-strength cement B is a brand the manufacture of which has since been discontinued.

Each cement was used with three different water contents: 4½, 6, and 9 gal. per sack of cement. The mix was varied with each water content to give a constant consistency—approximately 3 to 4-in. slump. The concrete mixes are given in the notes accompanying Fig. 1.

Oven-dried sand and gravel from Elgin, Ill., were used for the concrete and mortar. The aggregates were graded from 0 to 1½-in. for the concrete strength and durability specimens, and from 0 to

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No. 4 for the mortar bars for length change measurements. For the capillary flow specimens mine-run Ottawa silica sand was used.

Elgin coarse aggregate contains some non-durable particles—chert, so-called "chocolate bars" and other soft particles. Since the presence of these non-durable particles would tend to confuse the results of the freezing-and-thawing tests, for the specimens exposed to alternate freezing, the coarse aggregate was hand-picked to remove most of the non-durable material.

TABLE I.—CHEMICAL ANALYSIS. COMPOUND COMPOSITION AND FINENESS OF CEMENTS.

(A) CHEMICAL ANALYSIS, PER CENT BY WEIGHT

Each value is the average of two determinations.

	Silica (SiO_2)	Iron Oxide (Fe_2O_3)	Aluminum Oxide (Al_2O_3)	Calcium Oxide (CaO)	Magnesium Oxide (MgO)	Sulfuric Anhydride (SO_3)	Loss on Ignition	Insoluble	Free
								Residue	CaO
Normal portland cement....	21.19	2.79	5.56	63.55	3.09	1.64	1.18	0.13	0.24
High-early-strength cement A	19.68	2.22	5.87	66.26	1.38	2.40	1.46	0.11	1.11
High-early-strength cement B	18.31	2.95	7.79	65.90	0.87	2.14	1.54	0.12	1.08

(B) COMPUTED COMPOUND COMPOSITION FROM ANALYSIS, PER CENT BY WEIGHT

	C_3S	C_2S	C_3A	C_4AF	$CaSO_4$
Normal portland cement....................	50.4	22.8	10.0	8.5	2.8
High-early-strength cement A..................	66.0	6.8	11.8	6.7	4.0
High-early-strength cement B..................	62.0	5.9	15.6	9.0	3.6

(C) FINENESS

Percentage passing No. 200 sieve determined in accordance with the Standard Specifications and Tests for Portland Cement of the American Society for Testing Materials.

Each value is the average of two tests.

	Percentage Passing a No. 200 Sieve	Air Analyzer, per cent by weight finer than size indicated			
		Tube A, 0.0017 in., 45 μ	Tube B, 0.0011 in., 30 μ	Tube C, 0.0007 in., 20 μ	Tube D, 0.0004 in., 10 μ
Normal portland cement.................	86.4	67.8	51.2	36.0	23.2
High-early-strength cement A.............	98.9	89.6	76.8	58.5	34.7
High-early-strength cement B.............	98.5	86.5	74.4	56.9	34.5

The freezing-and-thawing tests were made on 6-in. concrete cubes. The cubes were completely submerged in water in individual cans and piled in layers 3 cans high on lift trucks and placed in the refrigerator for about 20 hr. During this period the temperature of the refrigerator was lowered from about +10 F. (−12 C.) to about −15 F. (−26 C.). At the end of the 20-hr. period the cubes were removed from the refrigerator and thawed in water at about 80 F. (27 C.). This freezing-and-thawing period is referred to as 1 cycle.

Mortar bars, 3 by 3 by 15 in., were used for length-change measurements. The measurements were made by means of a fulcrum plate strain gage on brass plugs inserted in the top surface of the specimens at time of molding.

The capillary flow test and the apparatus used were designed by Raymond Wilson and C. L. Ford of the laboratory staff. The apparatus (see insert in Fig. 5) consisted essentially of a glass bottle containing a $\frac{7}{8}$ by 5-in. mortar specimen projecting through a snugly fitting rubber dam which prevented any loss of water except through the specimen.

TABLE II.—EFFECT OF ALTERNATE FREEZING AND THAWING ON STRENGTH.

Strength results for moist curing were obtained from age-strength curves of 6 by 12-in. concrete cylinders shown in Fig. 1 for ages corresponding to the ages of the cubes.

Strength results for specimens frozen and thawed were obtained from 6-in. concrete cubes. The results reported are 85 per cent of actual test values in order to place them on the same basis as 6 by 12-in. cylinder strengths.

Values in parentheses are strength ratios based on the strengths of the moist-cured specimens of same age.

	Original Curing Period, days	At First Sign of Disintegration				Tests at Later Period[a]			Tests at Later Period[b]		
		Number of Cycles	Compressive Strength, lb. per sq. in.		Age at Test, months	Compressive Strength, lb. per sq. in.		Age at Test, months	Compressive Strength, lb. per sq. in.		Age at Test, months
			Freezing-and-Thawing Specimens	Moist Cured		Freezing-and-Thawing Specimens	Moist Cured		Freezing-and-Thawing Specimens	Moist Cured	
WATER CONTENT—6 GAL. PER SACK OF CEMENT											
Normal portland cement...........	15	60	4180 (77)	5450	3	4250 (73)	5800	5	4470 (77)	5800	6
High-early-strength cement A.........	3	180	4550 (75)	6050	9	4130 (68)	6100	11	4430 (71)	6200	12
High-early-strength cement B.........	7	240	3720 (68)	5450	11	4480 (82)	5450	11	1790 (32)	5550	14
WATER CONTENT—9 GAL. PER SACK OF CEMENT											
Normal portland cement...........	28	60	1130 (41)	2750	3	Disintegrated	....	..	Disintegrated	....	..
High-early-strength cement A.........	3	40	2400 (67)	3600	3	2400 (67)	3600	4	2620 (72)	3650	5
High-early-strength cement B.........	4	90	2400 (92)	2600	6	2100 (75)	2800	9	2250 (80)	2800	9

[a] Represents 50 cycles more than at first sign of disintegration.

[b] Represents 60 cycles more than at first sign of disintegration.

PRINCIPAL RESULTS OF THE TESTS

The test results are shown in Tables I and II and in Figs. 1 to 5. Table I gives the chemical analysis, compound composition and fineness of the cements. Table II contains the strengths of the cubes tested after different time exposures to alternate freezing and thawing.

Strength Tests.—In the lower set of diagrams in Fig. 1 are water-cement ratio - strength curves for 6 by 12-in. moist-cured concrete cylinders at ages of 1 day to 2 years. All the cements gave water-cement ratio - strength curves similar in shape to those obtained by other investigators for moist-cured concretes. For a given water-cement ratio, the 1, 3, 7, and 28-day strengths for the high-early-strength cements were higher than those obtained with the normal portland cement. At ages of 1 and 2 years the strengths are about the same for both types of cements.

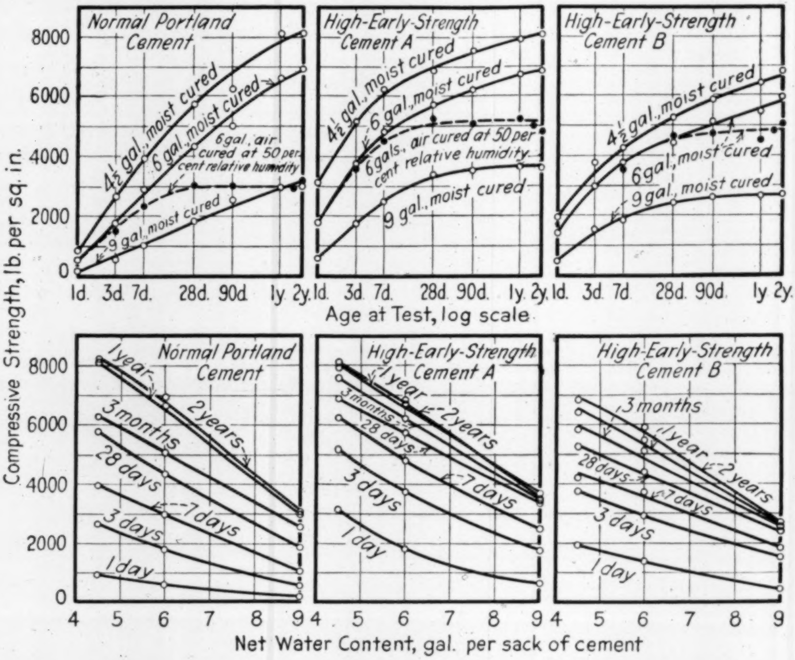

FIG. 1.—Compressive Strength of Concrete Made with High-Early-Strength and Normal Portland Cement.

Compression tests of 6 by 12-in. concrete cylinders.

Unless otherwise noted, specimens were moist cured until test; tested wet.

Mix by weight: $4\frac{1}{2}$-gal. concrete, 1:1.64:3.02; 6-gal. concrete, 1:2.53:3.80; 9-gal. concrete, 1:4.36:5.41.

In the upper set of diagrams in Fig. 1 are age-strength relationships for moist-cured concrete of three different water contents, $4\frac{1}{2}$, 6, and 9 gal. per sack, and also for a 6-gal. concrete which was cured in air at 50 per cent relative humidity after 1 day in the molds. The moist-cured specimens in these groups were tested wet, the air-cured specimens were tested dry.

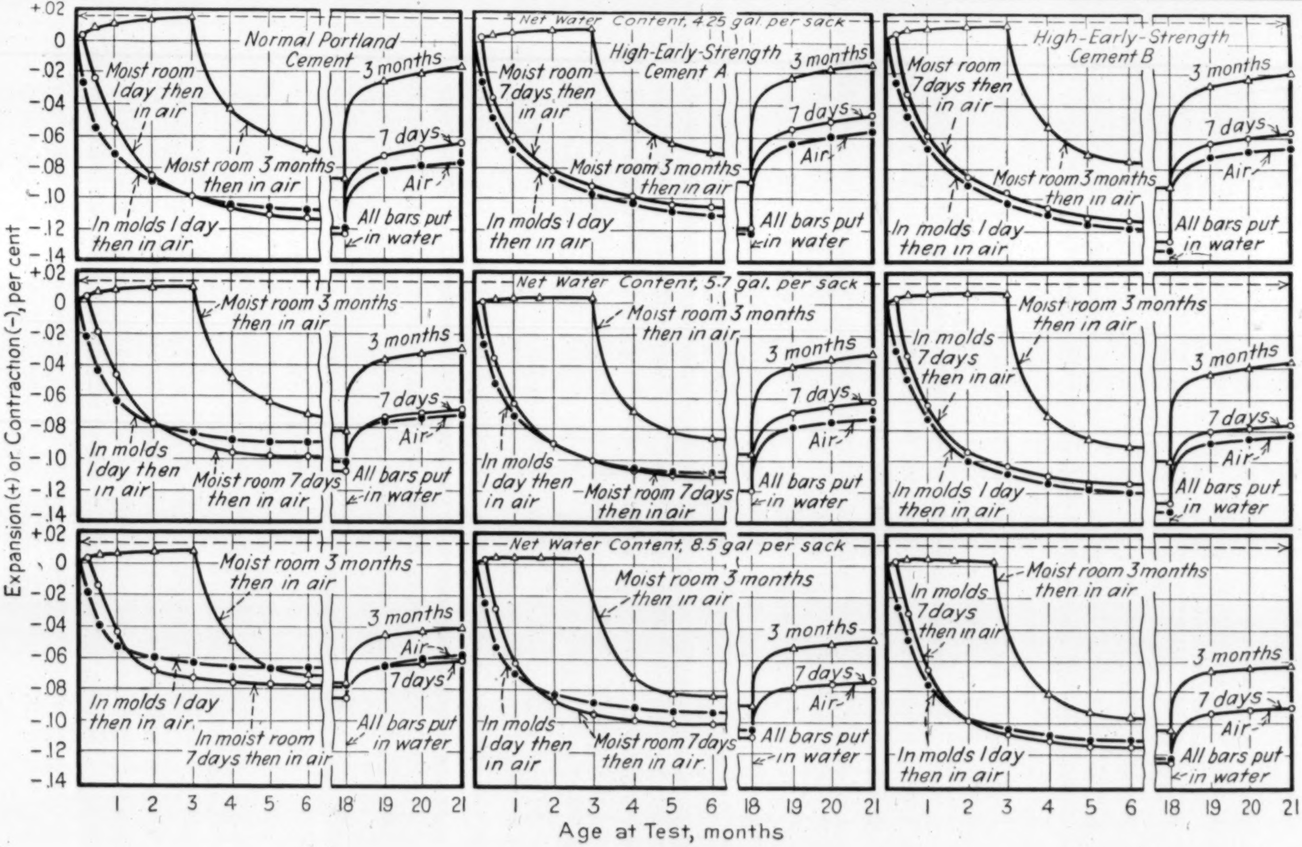

FIG. 2.—Volume Change of Mortars.

3 by 3 by 15-in. mortar bars. Aggregate: 0 to No. 4 Elgin sand. Mix by weight: 4.25-gal. mortar, 1:1.75; 5.7-gal. mortar, 1:2.94; 8.5-gal. mortar, 1:4.77.

This set of diagrams brings out no new features in the comparison of the two types of cements except that in the case of the high-early-strength cements the differences between the strength of moist and air-cured specimens at ages greater than 28 days are much smaller than for the normal cement. This indicates that a short period of moist curing develops a large proportion of the potential strength of the high-early-strength cements. The fact that the air-cured specimens were tested dry makes the results somewhat higher than they would have been if the air-cured concrete had been tested wet.

Strength specimens are on hand for test at the 5 and 10-year periods for all conditions.

Length Change Measurements.—Figure 2 presents data on changes in length observed on 3 by 3 by 15-in. mortar bars. Initial readings were taken at 1 day. Thus length changes during the plastic period are excluded. Three periods of moist curing were used: 1 day, 7 days, and 3 months, followed by storage in the air of the laboratory at 50 percent relative humidity for periods up to 18 months, at which time the specimens were placed in water. For each of the above conditions 3 water contents were used with each type of cement. In Fig. 2, the diagrams are shown broken between the ages of 6 and 18 months in order to permit the use of a larger scale. The shrinkage between 6 and 18 months was relatively small. All comparisons of shrinkage are based on total shrinkage, that is, the contraction between the point of greatest expansion at the end of the moist-curing period and the shrinkage at the 18-months period.

The mortars expanded when cured in the moist room and shrank when exposed to the air. The magnitude of the expansion from the original condition as compared to the shrinkage in the air of 50 per cent relative humidity is negligible. The following tabulation shows the expansions at 3 months:

	Expansion at 3 months for Mortars of Different Water Contents, per cent		
	4.25 gal. per sack	5.70 gal. per sack	8.50 gal. per sack
Normal cement	0.016	0.012	0.008
Cement A	0.008	0.004	0.004
Cement B	0.012	0.006	0.004

As may be seen from the tabulation, the greatest expansion at 3 months was obtained with the normal cement and the least with the high-early-strength cements. Reference to the figure shows that while in all cases expansion increases with duration of moist curing the rate of increase at 3 months is extremely small.

The total shrinkage at the 18-month period for the 7-day and 3-month moist-cured mortars in Fig. 2 were:

	Total Shrinkage at 18-month Period for Mortars of Different Water Contents, per cent					
	7-day Moist Cured			3-month Moist Cured		
	4.25 gal. per sack	5.70 gal. per sack	8.50 gal. per sack	4.25 gal. per sack	5.70 gal. per sack	8.50 gal. per sack
Normal cement	0.130	0.112	0.089	0.101	0.092	0.085
Cement A	0.122	0.121	0.112	0.098	0.099	0.094
Cement B	0.131	0.131	0.128	0.103	0.107	0.107

The shrinkage of the mortars cured 1 day in molds and the mortars cured 7 days in the moist room were very similar and the results of the length change measurements for the 1-day curing were omitted from the tabulation. In general, greatest shrinkage was obtained with high-early-strength cement B and least shrinkage with the normal cement. The difference in shrinkage between the different types of cement was not great, except in the case of the 8.5-gal. mortars.

An important observation from these data is the reduced shrinkage with continued moist curing beyond 7 days. This was evident regardless of the water content or type of cement and was most noticeable for the mortars made with a water content of $4\frac{1}{4}$ gal. per sack of cement. The fact that the mortars of lower water content are affected more by increased curing is logical, since they contain greater volumes of paste, and any factor affecting the paste will be reflected in the behavior of the mortar to a greater degree the greater the quantity of paste. Even at 18 months the mortars had not reached equilibrium. However, the rate of loss of water was so small that any conclusions based on the 18-month measurements would not be affected by the slight additional shrinkage that might occur before equilibrium is reached.

After the 18-months drying period, the mortar bars were put in water and expansion measurements made for 3 additional months. In general, the bars with no original curing and those cured 7 days moist expanded about the same when placed in water, while bars cured 3 months moist showed slightly greater expansion. This appears to be true regardless of type of cement or water content.

Absorption.—Figure 3 shows the absorption of water by the mortar bars following their return to water storage after 18 months of drying. The absorption is expressed as a percentage of the volume of cement paste in the mortar. Only the normal cement and high-early-strength cement A are shown in this figure.

Several interesting features are brought out by the comparisons offered in these diagrams. In all cases the absorption was less for improved quality whether brought about by lower water-cement ratio or longer moist-curing period. Considering the absorption for the first 5 or 6 hr. of immersion, the effect of the long period of curing was more pronounced in the leaner mixes, while for the 28-day absorption the richer mixes were improved the most. In all cases the more rapid hardening cement showed the least absorption but the difference was less important for the longer curing period.

The reduction in absorptiveness brought about by the improved quality of paste through lower water content or better curing is consistent with the behavior of the specimens subjected to alternate freezing and thawing as will be shown in the following section. The improved water-tightness combined with the greater strength due to better curing develops the improvement in resistance to the destructive effect of the repeated freezings.

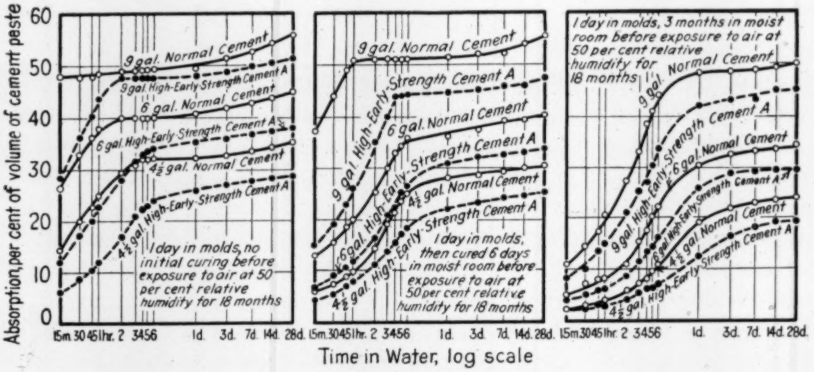

FIG. 3.—Absorption of Mortar.

Freezing-and-Thawing Tests.—Freezing-and-thawing data up to 360 cycles are shown in Fig. 4. These durability studies were made on 6-in. concrete cubes which were introduced into the refrigerator at time periods so arranged that the concrete had equal strengths at the start of the freezing-and-thawing cycles. For the 6-gal. concrete, four strengths were used: 1750, 3800, 4900, and 5950 lb. per sq. in. For the 9-gal. concrete the strengths were 1750, 2450, 3350, and 3550 lb. per sq. in. Under this method of testing, the concretes from different cements were of different ages when exposed to the action of freezing and thawing. The respective ages are shown on the various graphs in Fig. 4.

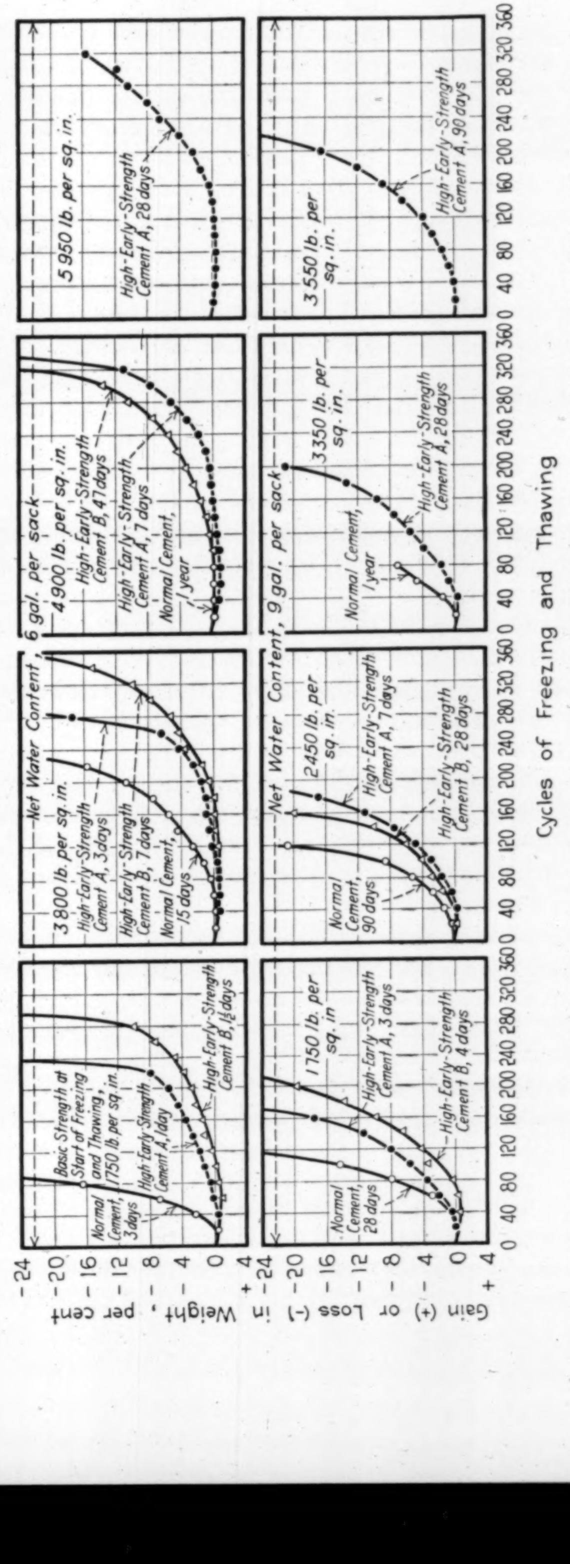

FIG. 4.—Freezing-and-Thawing Tests of 6-in. Concrete Cubes Moist Cured for Various Periods Before Exposure.

Mix by weight: 6-gal. cubes, 1:2.53:3.80; 9-gal. cubes, 1:4.36:5.41.

In the richer mix, 6 gal. per sack, moist curing up to about 2 weeks improved resistance to disintegration, but in the 9-gal. mixes prolonged curing gave virtually no improvement. Unlike the improvement effected in the case of strength, the resistance seems to reach a maximum beyond which no further improvement is shown by continued moist curing. This is clearly brought out in Fig. 4 by the curves for high-early-strength cement A in the upper group of diagrams where the maximum resistance was reached after 7 days curing. It is also shown for the normal cement in the 9-gal. concrete where curing up to a period of 1 year before exposure did not increase the resistance to freezing and thawing over that shown for the 28-day curing period.

In considering these data the fact should not be overlooked that some additional curing is obtained during the early exposure period. The specimens are at all times submerged in water and during the thawing period, at least (4 to 6 hr. every day), to moderate temperatures. The high-early-strength cement seems to have an advantage in this respect, due no doubt to its more rapid rate of hardening in the early curing period.

This effect of additional curing makes it difficult to obtain a direct comparison between the two types of cements exposed to freezing action when a given strength is attained. However, certain comparisons are of interest. For the specimens exposed when a strength of 1750 lb. per sq. in. was attained, the richer mixes (6 gal.) of the high-early-strength cements cured 1 and $1\frac{1}{2}$ days showed distinctly better resistance than the leaner mixes (9 gal.) cured respectively 3 and 4 days. But with the normal cement, the rich mixes cured 3 days before exposure were slightly less resistant than the lean mix cured 28 days.

No comparison exactly parallel to the above is offered for the higher strength concretes, but the 6-gal. mixes introduced into the refrigerator at a strength of 3800 lb. per sq. in. can be fairly compared with the 9-gal. mixes of strengths 3350 and 3550 lb. in the two right-hand diagrams of the lower group. For both types of cements the richer mixes of shorter curing showed a greater degree of resistance over the leaner mixes cured longer periods, than is indicated by the small difference in strength at the time of exposure. It is interesting to compare also the richer mixes of 1750-lb. strength at the time of exposure with the leaner mixes of 2450-lb. strength. For the high-early-strength cements the richer mixes again showed a decided advantage even though they had a disadvantage of 28 per cent less strength at the time of exposure. For the normal cement, the leaner mix cured 90 days (2450-lb. strength) was definitely better than the rich mix cured only 3 days (1750-lb. strength). It appears from this that for the normal cement the additional curing during the alternate freezing and thawing, when exposed at 3 days, is not sufficient to develop greatly improved resistance. While the data offer no comparison it is probable that with a few more days early curing a comparison for the normal cements would be of the same order as for the high-early-strength cements.

For each set of cubes used in determining the curves in the second diagram for the 6-gal. concrete and the first diagram for the 9-gal. concrete in Fig. 4 there was a companion set of 3 cubes which were introduced into the refrigerator at the same time and cured through the same number of cycles until the first signs of disintegration were evident, at which time one of the extra 3 was tested in compression. After another 50 cycles the second cube was tested and after another 10 cycles the third cube. The strengths of these cubes are given in Table II. The table shows the age and strength of the specimens when removed from the test, also the strengths of similar concretes cured moist for the same period of time. The values shown for the cube strengths are the actual test results reduced by 15 per cent to give the equivalent strength in terms of 6 by 12-in. cylinders.

In all cases the concrete exposed to freezing and thawing shows lower strength than similar concrete moist cured for the same age. For the 6-gal. concrete the strength at the time of the first signs of disintegration was 68 to 77 per cent of the strength of similar moist-cured concrete of the same age. In the case of the 9-gal. concrete the strength ranged from 41 to 92 per cent. The 41-per-cent value was obtained with the normal portland cement. This low strength for the normal portland was reflected in the rapid disintegration after the first signs were noted. In general, the strength decreased with additional exposures for 50 and 60 cycles. It is worthy of note that in all but one case the strength of the specimen tested at first sign of disintegration was greater than the strength at the time of exposure to the repeated freezings.

Capillary Flow Test.—In order to study further the rate at which cement pastes harden in water and thus bring out the effect of early curing and differences in types of cement, a new type of test was tried. This test, which has been termed the capillary flow test, consists in measuring the amount of water evaporated from a specimen, one end of which is immersed in water. The apparatus used is sketched in Fig. 5 on which are plotted also the results of tests in terms of the average daily flow against the age of the specimen.

Separate curves are shown for 3 different mortar mixes for each of the two types of cement. The mixes were made with water contents of 50, 65, and 80 percent by weight of cement (equivalent to 5.6, 7.2, and 9.0 gal. per sack, respectively) with sufficient mine-run Ottawa sand added to give a plastic consistency suitable for molding in the $\frac{7}{8}$-in. diameter by 5-in. specimens.

This type of test was found to be particularly valuable in comparing the permeabilities at early ages, and in showing the effect of type of cement upon the development of increased water-tightness during the early hardening period. For the mortars shown in this figure, with 50 and 65 per cent water by weight of the cement, the specimens were placed in the flasks and exposed to a blast of air from an electric fan after curing only 1 day in the molds. For the specimens with 80 per cent mixing water, a longer time in the mold was necessary to avoid injury in handling. Since the specimens were partly submerged in the flask and only the upper end exposed to the drying atmosphere the curing continued much the same as if in an all moist condition. The effect of this continued curing is shown in the reduced daily flow for all of the mortars indicated on the figure. An interesting point in this test, not brought out by Fig. 5, was that the flow at any particular age of the specimen was the same whether the specimen was placed in the flask on that particular day or had been in the flask and subjected to the capillary effect prior to

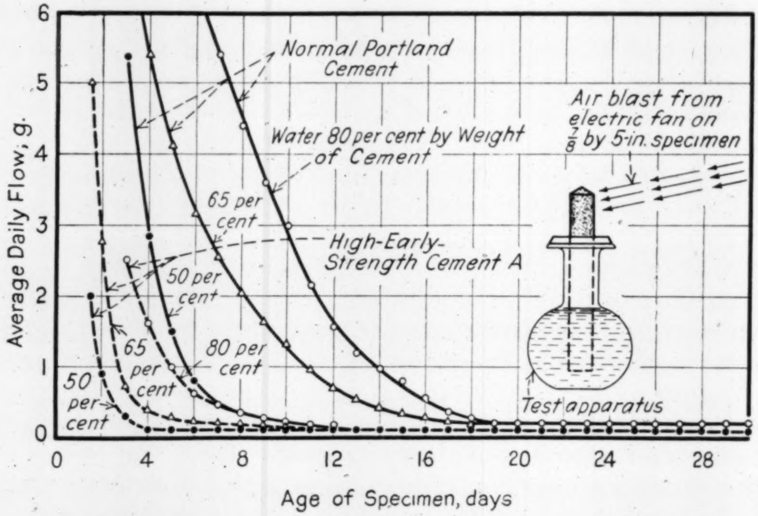

FIG. 5.—Capillary Flow as Influenced by Continuous Moist Storage.

that time. All of the points represented on Fig. 5 are for the specimens introduced at the age of 1 or 2 days as mentioned above.

It will be noted from the curves that the permeabilities of the mortar decreased rapidly at first, then more slowly, finally reaching a uniform rate which apparently does not change over a relatively long period of time. The effects of the lower water ratio with both cements and the increased rate of action in the case of the high-early-strength cement in bringing the specimen to the condition of constant flow at an early period are clearly seen. For example, the specimen of high-early-strength cement and 50 per cent mixing water obtains at the second day a rate of flow which was not reached until the fourteenth day by the specimen of normal portland cement mixed with 80 per cent water.

While the data from these tests cannot be directly compared with the other results reported herein, they do show the direct effect of increased curing on the development of water-tightness. They also show the effect of the more rapid hardening in the high-early-strength cements on the early development of a given degree of water-tightness.

Inasmuch as the richer mortars reached a constant rate of capillary flow within 2 weeks, this may be taken as indicating the reason why prolonged curing of the richer mix did not increase resistance to freezing and thawing as discussed above.

SUMMARY

This investigation was undertaken to supply information on the effect of moist curing on strength, volume change, absorption, durability, and permeability as measured by capillary flow. Both normal portland and high-early-strength cements were investigated.

Following are the principal results:

1. Typical water-cement ratio - strength curves for moist-cured concrete were obtained regardless of the type of cement or test age. A significant feature of the strength tests was the smaller differences between the strength of moist-cured and air-cured specimens at ages greater than 28 days exhibited by the high-early-strength cement.

2. Increased expansion of mortars in moist air but decreased shrinkage in dry air at 18 months resulted from longer periods of moist curing. This was true for all water contents and for both types of cement. The difference in volume change between the normal portland and high-early-strength cement was not of great importance.

3. Mortars of better quality were less absorptive, regardless of

whether the better quality was due to a lower water content or prolonged curing.

4. Up to a certain period, increasing the duration of moist curing of the concrete before exposure increased its resistance to the destructive effects of freezing and thawing, but further curing had no effect in increasing resistance. The length of this beneficial curing period depended on the type of cement and water content of the paste. With a given cement and curing periods sufficient to develop the maximum resistance in each case, the richer mixes gave greater resistance than lean mixes of the same strength at the time of exposure.

5. The rate of capillary flow in mortars is influenced by duration of exposure, by type of cement and quantity of mixing water. Even the mortars of high water content in a relatively short time reached a uniform rate of leakage which was quite small as compared with the rate of leakage when first exposed.

DISCUSSION

Mr. M. O. Withey[1] (*presented in written form*).—This paper contains much valuable data on the effect of duration of moist curing on the strength and absorptive properties of concrete made with the principal types of portland cement.

The data indicate that concrete made with the high-early-strength cement A was superior in strength at early ages and required a shorter period of moist curing for a given strength than concrete of like water content made with the normal portland cement. The lower absorption of the concrete made with the high-early-strength cement and the markedly superior resistance to freezing and thawing as evidenced by the strength ratios in Table II of the paper of concretes of the high-early-strength cements is information of much importance to those interested in winter construction.

It would be of interest to know whether the volume changes of the mixes shown in Fig. 2 correspond to the relationship which was worked out by Mr. Lagaard some years ago at the Association Laboratory.

In Table II and Fig. 4 data are given showing the resistance to freezing and thawing. These data, like some of our own, indicate that the loss in weight is far from being a quantitative measure of the loss in strength. For example, according to the data on concrete made from high-early-strength cement A, a loss of strength of 32 per cent for a concrete with a moist-cured strength of 6100 lb. per sq. in. was accompanied by a loss in weight of only 1 per cent. Likewise, for a concrete of normal portland cement exhibiting 5800 lb. per sq. in. moist-cured strength, a 27 per cent loss in strength was accompanied by a loss in weight of only 1 per cent. In another instance, the concrete made from the normal cement cured 28 days showed a 59 per cent loss in strength after 60 cycles of freezing and thawing whereas the loss in weight was only 3 per cent. These examples indicate very clearly that a very large loss in strength may be accompanied by only a slight change in weight and little visible evidence of disintegration. Hence, the shapes of the curves in Fig. 4 are not characteristic of curves representing loss in strength. It is to be hoped that the author may later be able to supply similar data based on loss in strength.

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[1] Professor of Mechanics, University of Wisconsin, Madison, Wis.

The latter part of conclusion No. 4 of the paper indicates that with a given cement and a sufficient time of curing to develop maximum resistance to freezing and thawing, the richer mixes were more resistant than the leaner mixes of the same strength. This statement should not be misinterpreted to mean that richer mixes of a given strength are more durable than lean mixes of the same strength and curing conditions.

MR. C. A. HUGHES[1] (*by letter*).—That moist curing increases the resistance to freezing and thawing during early ages is well established by the author's data and by those of other investigators. However, there is much yet to be learned about the effect of continued curing. The need for such information is increased by the fact that available data apparently do not correlate with accepted ideas as to the effect of continued hydration on strength and porosity. Since disintegration due to frost action results chiefly from the pressure of ice forming internally, it seems to follow that increased resistance to freezing and thawing would result from increased strength and decreased porosity.

TABLE I.—APPROXIMATE NUMBER OF CYCLES OF FREEZING AND THAWING AT LOSSES OF 8 AND 20 PER CENT BY WEIGHT.

Cement A			Cement B			Normal Cement		
Initial Curing Period, days	Number of Cycles		Initial Curing Period, days	Number of Cycles		Initial Curing Period, days	Number of Cycles	
	8 per cent loss	20 per cent loss		8 per cent loss	20 per cent loss		8 per cent loss	20 per cent loss
WATER-CEMENT RATIO, 6 GAL. PER SACK								
1	220	236	1½	270	295	3	65	85
3	265	283	7	300	355	15	180	230
7	305	330	47	265	318			
28	260	340[a]						
WATER-CEMENT RATIO, 9 GAL. PER SACK								
3	120	165	4	145	200	28	80	110
7	140	185	28	130	160	90	95	119
28	145	200				365	85[a]	
90	155	210						

[a] Extrapolated.

The author's data show both increased strengths and decreased porosities with continued curing, though the capillary flow tests indicate little if any decrease in porosity after the first three weeks even for lean mixes. Assuming criterions of the resistance to freezing and thawing as the numbers of cycles at 8 and 20 per cent loss, respectively, the approximate data of the accompanying Table I was read from Fig. 4 of the paper.

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[1] Associate Professor of Structural Engineering, University of Minnesota, Minneapolis, Minn.

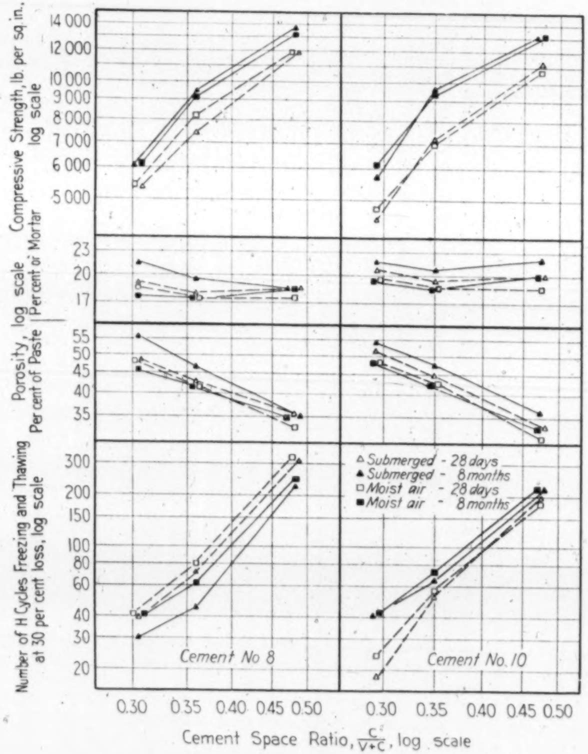

FIG. 1.—Compressive Strengths, Porosities and Resistances to Freezing and Thawing of 2-in. Mortar Cubes. Each point represents the average for three specimens.

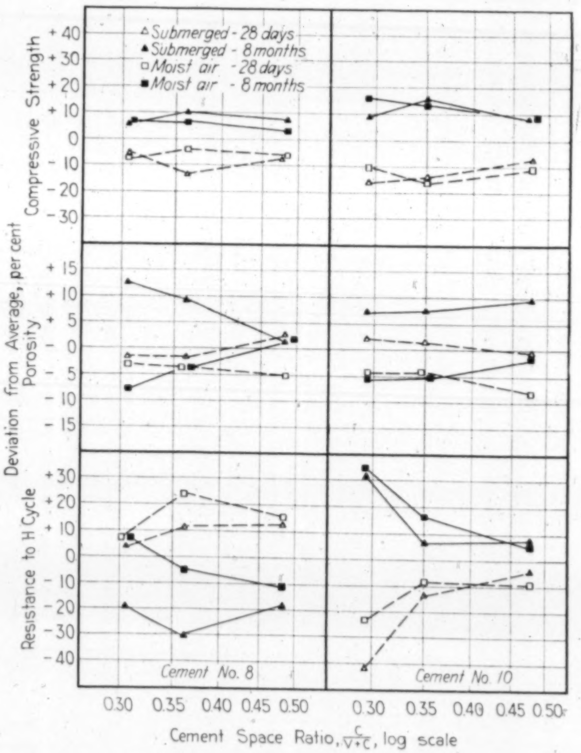

FIG. 2.—The Data of Fig. 1 Expressed as Percentage Deviations from the Average of All Specimens of the Same Mix.

If the above criterions be accepted, then it is evident that prolonged curing not only did not increase the resistance, but decreased it in certain cases. It is true that the decreases shown are relatively small and may well be due only to the variations that are bound to occur in a test as difficult to control as freezing and thawing. Nevertheless, further tests should be made to establish whether or not such decreases are indicative of reduced resistance at later ages. The writer has recently obtained some data which he hopes may be of interest as a supplement to that presented by the author.

TABLE II.—STRENGTHS, PASTE POROSITIES AND RESISTANCES TO FREEZING AND THAWING OF SUBMERGED SPECIMENS RELATIVE TO MOIST AIR-CURED SPECIMENS. AGE CONSTANT.

Age	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight
	Cement Space Ratio, $\frac{c}{v+c} = 0.483$			Cement Space Ratio, $\frac{c}{v+c} = 0.360$			Cement Space Ratio, $\frac{c}{v+c} = 0.304$		
Cement No. 8:									
28 days	−0.17	+7.28	−1.81	−8.80	+1.87	−12.38	+1.25	+1.03	−2.64
8 months	+4.42	+0.58	−6.87	+3.50	+12.62	−24.68	−0.52	+20.69	−26.81
	Cement Space Ratio, $\frac{c}{v+c} = 0.479$			Cement Space Ratio, $\frac{c}{v+c} = 0.349$			Cement Space Ratio, $\frac{c}{v+c} = 0.295$		
Cement No. 10:									
28 days	+3.69	+7.51	+6.00	+2.18	+6.18	−4.95	−4.99	+6.97	−19.20
8 months	0	+10.52	+0.50	+2.13	+12.05	−9.73	−7.30	+12.34	−3.20

TABLE III.—STRENGTHS, PASTE POROSITIES AND RESISTANCES TO FREEZING AND THAWING OF 8-MONTH SPECIMENS RELATIVE TO 28-DAY SPECIMENS. CURING CONDITION CONSTANT.

Curing Condition	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight	Compressive Strength	Porosity of Paste	Number of H cycles at 30 per cent Loss in Weight
	Cement Space Ratio, $\frac{c}{v+c} = 0.483$			Cement Space Ratio, $\frac{c}{v+c} = 0.360$			Cement Space Ratio, $\frac{c}{v+c} = 0.304$		
Cement No. 8:									
Moist air	+9.37	+6.12	−26.82	+11.03	0	−29.44	+13.13	−5.27	0
Submerged	+14.06	−0.58	−31.88	+23.33	+10.75	−41.74	+11.36	+14.39	−24.17
	Cement Space Ratio, $\frac{c}{v+c} = 0.479$			Cement Space Ratio, $\frac{c}{v+c} = 0.349$			Cement Space Ratio, $\frac{c}{v+c} = 0.295$		
Cement No. 10:									
Moist air	+19.72	+6.91	+16.53	+29.12	−0.21	+25.80	+26.44	−0.44	+57.60
Submerged	+16.03	+9.92	+11.03	+29.07	+5.66	+21.02	+24.13	+4.97	+73.60

In a paper presented before this Society in 1933,[1] it was shown that the resistance of mortar cubes cured 90 days submerged in water was less than when stored 14 days in water, 11 days in air and 3 days in water, prior to the first cycle of freezing and thawing. Data from another investigation indicated that this unexpected result was probably attributable to the period of air curing. It was suspected, however, that the relatively low resistance of the 90-day specimens might be partly due to leaching during storage under water. Consequently, the two portland cements showing the greatest and least relative resistance at 90 days were used in a short series, in which half the specimens were submerged in water and half were cured in moist air for 28 days and 8 months, respectively, prior to test. The consistency was approximately constant for the three mixes used. The "porosities" are the differences in weight of the specimens surface dried on removal from the moist room and oven-dried expressed as percentages by volume of the mortar or of the paste. The freezing and thawing cycle was the *H* cycle described in the paper referred to.

The test values (averages of data from three 2-in. cubes) are shown in the accompanying Fig. 1, and the percentage deviations from the averages of all specimens of the same mix made from the same cement in Fig. 2. In the accompanying Tables II and III, the data are arranged to show more clearly the effect of the two curing conditions and the effect of the two ages respectively. These data are in good agreement with those of the original investigation except for the specimens of the richest mixes cured 90 days in water, the resistances of which were relatively low.

It is interesting to note from Fig. 1 that the porosities expressed in terms of the mortar volumes are substantially equal for all three mixes and therefore give no information as to the relative resistance to freezing and thawing. The correlation indicated between the paste porosity and the resistance to freezing and thawing is to be expected since both are functions of the cement-space ratio.

The porosities of the moist air cured specimens were about equal at both ages (see Fig. 1). That leaching occurred is plainly evidenced by the increased porosity of the specimens submerged in water, the increase being much greater at 8 months than at 28 days (see Table II).

Examination of Table II will show that for the same initial age, an increase in porosity was accompanied by a decrease in resistance to freezing and thawing, except for the richest mix of cement No. 10.

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[1] C. A. Hughes, "The Durability of Cement Mortars; the Cement and Method of Testing Major Variables." *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 511 (1933).

This is also more or less true for the specimens made from cement No. 8 when the data are compared with the curing condition constant and the age varying (see Table III). It will be noted from Figs. 1 and 2 that the resistance to freezing and thawing of the 8-month specimens of this cement was less than that of the 28-day specimens. In contrast, the resistance of the 8-month specimens of cement No. 10 was greater than that of the 28-day specimens. In this case increased resistance was obtained with increased porosity. Evidently some factor or factors other than porosity are involved. The writer has found no explanation for the difference in behavior of cements Nos. 8 and 10. It may possibly lie in their elastic properties or in the effect of over-all grading on the size and arrangement of pores. While the strength difference between the two ages is greater for cement No. 10 than for cement No. 8, this is believed to have little influence. The calculated constitutions for the two cements are so similar that no help is apparent from that quarter.

Since these cements were specially selected from 18, the contrast afforded by them after prolonged curing is greater than probably will attain for any other two cements not so selected. As might be expected, therefore, the results of the author, while exhibiting the same general characteristics, do not do so to the same degree, at least up to the ages for which data are given. Also, of course, the effect of leaching will be much more noticeable for 2-in. mortar cubes than for 6-in. concrete cubes.

MR. H. J. GILKEY[1] (*by letter*).—On page 335 of the paper, the author states: "An important observation from these data is the *reduced shrinkage with continued moist curing beyond 7 days*." The writer questions the validity of this statement both on the basis of the data presented in this paper and from tests performed by himself and others in 1928 and 1929.[2]

For the 4.25 gal. per sack mixtures (top of Fig. 2 of the paper) there is shown a small reduction in total shrinkage after the swelling that occurs in 3 months has been added to the shrinkage below the zero line. For example, the normal portland cement (upper left-hand box) swells 0.02 per cent and shrinks 0.09 per cent at 18 months total age. Thus, the volume change that occurs during the 15 months in air following the 3 months in the moist room is 0.11 per cent while that for the specimens remaining in the forms one day, as well as those which had one day in the moist room, is 0.12 per cent which is hardly a significant difference. It can be contended, of course, that the proper datum for judging shrinkage is the original volume (as out of mold) rather than the swollen volume after the period of moist curing. While this may be debatable for some cases, the writer inclines to the view that the change from the swollen volume is the important one for most structures.

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[1] Professor and Head, Department of Theoretical and Applied Mechanics, Iowa State College, Ames, Iowa.

[2] J. L. Savage, I. E. Houk, H. J. Gilkey and Fredrik Vogt, Engineering Foundation Arch Dam Investigation, Vol. II, p. 516, Fig. 164.

The case considered is the one for which the statement comes nearest being true but it does not represent the concrete of practice. A water-cement ratio of 4.25 gal. per bag (water-cement ratio = 0.56 approximately) can be used only with mortars or very rich concretes.

The concretes in the middle row of boxes have 5.7 gal. per sack (water-cement ratio = 0.75) which represent the richer of practical concretes. For the normal cement the status at 18 months is almost identical, the concrete cured for 3 months having a slightly smaller total shrinkage than that exposed at one day.

For the moderately wet or lean mixture, 8.5 gal. per sack (water-cement ratio = 1.14) the net shrinkage of the concrete cured for 3 months is identical with that for the one-day curing and the gross shrinkage exceeds it.

In these respects the writer's data[1] are similar to those shown. The mixture was that used in the Stevenson Creek model dam, maximum size of aggregate being ⅜ in. and water-cement ratio being 1.00 (7.5 gal. per sack). Specimens were exposed to the air of the laboratory at ages of 2 days, 7 days, 28 days, 3 months and 8 months and remained in air up to a total age of 342 days (virtually a year). At the termination of air exposure, the specimens exposed at 8 months were still shrinking at a fairly rapid rate while the others were almost dormant. The net shrinkage for the specimens exposed at 28 days and 3 months both slightly exceeded that for the 2- and 7-day exposures while the gross shrinkages were appreciably greater for the longer prior curings. The tabulation gives approximate values. Beams or bars 3 by 3 by 40 in. with end inserts were used:

Age Exposed	Swelling at Exposure, per cent	Net Change at 1 year, approximate, per cent	Gross Change at 1 year, per cent	After 24 hours Soaking at 1 year, per cent
2 days	....	−0.070	−0.070	+0.010
7 days	+0.005	−0.074	−0.079	+0.006
28 days	+0.012	−0.087	−0.099	+0.012
3 months	+0.015	−0.085	−0.100	+0.015
8 months	+0.018	−0.058[a]	−0.076[a]	+0.017

[a] 8 months specimens still shrinking at age of 1 year (almost 4 months in air).

NOTE.—To assist in visualizing these figures, 0.1 per cent = 1.2 in. per 100 ft.

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[1] J. L. Savage, I. E. Houk, H. J. Gilkey and Fredrik Vogt, Engineering Foundation Arch Dam Investigation, Vol. II, p. 516, Fig. 164.

It will be noticed that the numerical values in the tabulation are strikingly close to those shown in the paper except for the last column which represents the status after 24 hr. re-immersion at the termination of the period in air. Apparently the author's specimens recovered only about half the amount of those in the writer's tests. The writer's tests showed a close relationship between volume change and changes in weight. Changes in weight under air exposure, oven drying, re-immersion, etc., have been observed for many different mixtures and ages and the writer has yet to find a mixture that does not recover virtually its initial weight upon 24 hr. resoaking by immersion. This fact leaves him wondering whether the partial recoveries shown should be accepted as typical of concretes and mortars generally. Perhaps this point calls for further study before venturing a generalization.

The shapes of the resoaking curves for the 3-month curing (right-hand box of Fig. 3 of the paper) are typical of those obtained in the writer's tests.

Ample curing is so intimately related to all good qualities in concrete that prior to his own tests the writer believed that, of course, shrinkage would be decreased by increased curing. Disillusionment came with a piece of pavement on which every precaution was taken to avoid cracking by providing ample curing. Cracks were delayed but were not prevented. Both the author's and the writer's tests show that this was to be expected.

The writer desires to call attention to one other statement that he feels might give a misleading inference to some. The last sentence in paragraph 1 of page 334 states, "The fact that the air-cured specimens were tested dry makes the results *somewhat higher* than they would have been if the air-cured concrete had been tested wet." In this case the word *somewhat* probably means *not less than 20 per cent* and possibly 30 per cent or more. In spite of oft-repeated emphasis[1,2] upon the important differences in strength that accompany differences in test condition (dry or wet), there continues to be a tendency, on the part of many engineers, to overlook or under-rate this factor. Hence, the present insistence that "somewhat," as here used, represents much more than a negligible 5 or 10 per cent.

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[1] H. F. Gonnerman, "Study of Methods of Curing Concrete," *Proceedings*, Am. Concrete Inst., Vol. XXVI, pp. 359–396 (1930).

[2] H. J. Gilkey, "The Effect of Varied Curing Conditions upon the Compressive Strength of Mortars and Concretes," *Proceedings*, Am. Concrete Inst., Vol. XXII, pp. 395–431 (1926).

Discussion by H. J. Gilkey of Symposium on Field Control of the Quality of Concrete, *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 424 (1927)

Discussion by H. J. Gilkey of paper by C. L. McKesson, "Finishing and Curing of Concrete Roads," *Transactions*, Am. Soc. Civil Engrs., Vol. 91, pp. 155–167 (1927).

In closing, the writer wishes to commend the author upon the excellence of his paper and upon the great fund of useful data that have been condensed to brief and readily available form. The points discussed relate to but a small part of the paper's valuable contents.

MR. A. T. GOLDBECK.[1]—We have been doing some work in expansion and contraction and I would like to bring out one point which, to me, is rather interesting. We made up concrete specimens, put them in a moist room and began to take expansion measurements. We observed the usual expansion due to moisture and the usual shrinkage due to the drying out of that moisture upon exposure to dry air.

At the end of four months' drying, we again immersed the specimens and they immediately expanded and finally reached constant length at the end of 20 to 30 days. The temperature of the water in which the specimens were immersed was then raised from 70 to 135 F. (21 to 57 C.) and the specimens expanded due to the increased temperature. However, when the temperature of the water was again lowered to 70 F. (21 C.) it was found that the specimens did not come back to their original length. These cycles of temperature, generally of two days' duration and ranging from 70 to 135 F. (57 C.) were repeated 60 times and the length of each specimen, measured at 70 F. (21 C.), gradually increased.

This increase in length was not due to the effect of the water alone, for it will be recalled that all of the specimens had previously reached constant length when immersed in water at a temperature of 70 F. (21 C.). The residual increase in length due to the 60 cycles of temperature alternation was roughly 0.0005 in. per inch of length, whereas the expansion of the specimens due to moisture alone was roughly 0.0003 in. per inch of length. Thus it becomes quite evident that the growth in length of concrete due to the combined effect of moisture and alterations in temperature is far more important than the increase in length of concrete due to moisture alone.

Perhaps this phenomenon helps to explain the gradual closing up of expansion joints under certain conditions and perhaps, also, it explains the gradual increase in warping of concrete slabs at expansion joints. I feel that more work along this particular line would be profitable.

MR. R. E. DAVIS.[2]—A year or so ago, we made some tests on small slabs in the field as a part of the Bay Shore highway investigation south of San Francisco, in which we had imbedded in the bottom of each slab Carlson strain meters and in the top of each slab strain gage plugs, so that we could determine the changes in the dimension of both the top and the bottom. The slabs were under observation approximately a year. At the end of that time, the total expansion in the bottom of the slab considerably exceeded the expansion that took place in corresponding concrete which had been continuously immersed in water at 70 F. (21 C.) in the laboratory. We have also noticed in connection with durability tests consisting of alternating high and low temperature, and of wetting and drying, that the modulus of elasticity of the concrete after this treatment was very much less than for corresponding concrete that had been cured under moist conditions for the same period of time, perhaps indicating the existence of small fractures within the mass.

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[1] Director, Bureau of Engineering, National Crushed Stone Assn., Washington, D. C.

[2] Professor of Civil Engineering, In Charge of Engineering Materials Laboratory, University of California, Berkeley, Calif.

MR. F. H. JACKSON.[1]—Certain observations which I have made have indicated that very thorough curing by means of water during the early period is of primary importance in developing the maximum strength and durability of concrete paving slabs. The burden of evidence seems to indicate that the first three days of curing are the most important. In this connection, it seems to me a question whether the use of the so-called surface seal blankets placed over the top of the concrete slab after it is, say 24 hours old, is sufficient, in that they do not supply any moisture to the slab other than that which is already contained in it after the concrete is hardened. I question whether we get the maximum curing effect under such conditions. As you all know, a very large proportion of our concrete pavement slabs are cured in this manner at the present time; that is, some sort of a surface covering placed over the concrete to retain the moisture. The question arises whether this is sufficient and whether additional moisture should not be added to the surface at least during the first few days of its existence.

MR. A. G. TIMMS[2] (*author's closure by letter*).—Replying to Mr. Withey's question, the length changes reported were measured on 3 by 3 by 15-in. mortar bars which were considerably smaller than those used in the tests reported by Mr. Lagaard. Also, all of the specimens were made with mortar, while the majority of the specimens used in determining the relationship found by Mr. Lagaard were based on concretes. In general, the specimens made with high-early-strength cement were in close agreement with Mr. Lagaard's earlier relationships. However, the normal portland cement mortars lost more water with a consequent greater shrinkage for a given water-cement ratio. The differences correspond to an increase in cement of $\frac{1}{2}$ to $\frac{3}{4}$ sacks per cu. yd.

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[1] Senior Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[2] Assistant Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

In regard to Mr. Hughes' discussion, the author does not believe one is quite justified in selecting losses of 8 and 20 per cent as a basis of comparison, because sharp changes in direction of the curves in Fig. 4 occur at about 8 per cent. Furthermore, the small decreases in resistance pointed out by Mr. Hughes probably reflect the variations that are bound to occur in freezing-and-thawing results because of the difficulty of controlling the test.

The leaching that Mr. Hughes obtained in 2-in. cubes would not be comparable to any leaching that might occur in dense concrete mixtures. Mr. Hughes shows in the table more examples of decreases in resistance for the 6-gal. than for the 9-gal. concrete, which appears to be at variance with the idea that leaching due to continued wet storage is a serious factor in the resistance of concrete.

Replying to Mr. Gilkey's discussion, the following tabulation shows the ratio of the total shrinkage after 3 months moist curing to that after 7 days moist curing:

	4.25 GAL. PER SACK	5.70 GAL. PER SACK	8.50 GAL. PER SACK
Normal cement	0.79	0.82	0.96
Cement A	0.80	0.82	0.84
Cement B	0.79	0.82	0.84

As may be seen, the total shrinkage for 3 months moist curing was from 96 to 79 per cent of that for 7 days moist curing, depending on the water-cement ratio and type of cement. These values show some reduction in shrinkage with prolonged moist curing, but as Mr. Gilkey points out, this may under certain conditions have little practical significance.

PLASTIC FLOW OF CONCRETE UNDER SUSTAINED STRESS

By R. E. Davis,[1] H. E. Davis,[1] and J. S. Hamilton[1]

Synopsis

This paper describes laboratory tests made on plain concrete cylinders and reinforced concrete columns subjected to sustained compressive stress. The tests have been carried on in the Engineering Materials Laboratory of the University of California over a period of about seven years.

The test results show the influence upon plastic flow or time-yield of the following variables: (1) duration of loading, (2) moisture condition of storage, (3) moisture content of concrete, (4) temperature of storage, (5) gradation of aggregate, (6) mineral character of aggregate, (7) size of mass, (8) age at time of application of load, (9) intensity of sustained stress, (10) lateral restraint, and (11) reinforcement. The extent of plastic recovery upon release of sustained load is indicated. In the studies on reinforced concrete columns, the variation in stress, with time, between the concrete and the steel is determined.

In the paper are discussed fundamental causes of flow, the interrelation between plastic flow and shrinkage, and the conditions under which flow under compressive stress may be important in design.

Among the conclusions it is pointed out that (*a*) flow in plain concrete may continue for periods of more than seven years, although the major portion of the deformation takes place within a year or two after application of load, (*b*) flow in reinforced concrete columns may practically cease after about one year under load for dry-stored columns, and after a few months for wet-stored columns, (*c*) the major portion of plastic flow is apparently due to seepage of colloidal water from the cement gel, although it is believed that some deformation may take place due to viscous and crystalline flow, (*d*) flow is markedly influenced by moisture condition of storage, moisture content of concrete, age of concrete, grading and character of aggregate, intensity of stress, size of mass, and reinforcement, (*e*) due to flow, the stress in the steel in reinforced concrete columns may in time reach or exceed the yield strength of the steel, while the concrete stress may be reduced to a value approaching zero.

Introduction

It is a recognized fact that plastic flow, or time-yield, takes place in concrete structures under stress. Tests have shown that, although the rate of flow decreases with time, this yielding continues for several years and may reach a considerable magnitude. Observations of reinforced-concrete columns over a period of years have indicated that due to flow there is a marked redistribution of stress between the concrete and the steel, such as to require consideration in design. There may be a wide variation in the magnitude of flow, depending upon the character of the concrete, upon the atmospheric conditions to which the concrete is exposed, and upon the size of the mass. With increasing refinement in the design of structures, the necessity for fuller information regarding the nature and limits of flow in concrete becomes obvious.

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[1] Professor of Civil Engineering, Instructor in Civil Engineering, and Research Assistant in the Engineering Materials Laboratory, respectively, University of California, Berkeley, Calif.

The purpose of this paper is to present the results of flow tests which have now extended over a period of seven years, to discuss fundamental causes of flow, and to point out conditions under which flow is important in design. While the investigation is principally concerned with axial flow under uni-directional sustained compressive stress, some tests on flow under triaxial loading conditions are reported.

Phenomenon of Flow:

A study of available data indicates that the flow of concrete under continued stress is due to no single cause, but to a combination of causes. It is probably impossible to tell definitely how much of an observed deformation is due to each of the influencing factors, but it is apparent that their relative importance will vary with the conditions to which the concrete is subjected.

In general, there appear to be three ways in which continuous yielding may take place. First, there may be a movement of particles one over the other, as in the flow of oil, asphalt, glue, or wet clay; this may be called *viscous flow*. Second, in a mass composed of crystals, slip may occur along planes within the crystal. This type of yielding, which in metals is called "creep," will be termed *crystalline flow*. The third way in which yielding may occur is by an actual loss of liquid from a mass composed of a liquid and a finely divided solid; this action will be called *seepage*, the term used to describe the phenomenon in colloidal systems.

That the yielding of concrete may involve all three kinds of flow is apparent from a consideration of the elements that make up a concrete mass. The major portion of the mass is composed of relatively chemically inert aggregate, usually a crystalline material. Surrounding each piece of aggregate is the chemically active cement-water paste, which in the hardened state binds the mass together. The cement, during hydration, is generally considered to form a gel, although in time this gel may be partly transformed into a crystalline state. Water may exist within the mass in three forms: (*a*) as chemically combined water, (*b*) as water which has been adsorbed by the cement colloids, called here *colloidal water*, and (*c*) as "free" water within the pore spaces or voids.

The function of the water in relation to time-yield and other volume changes which take place within the mass has been well explained by Lynam.[1] He gives evidences that the free water plays no direct part in volume changes. He also indicates that the process of hydration is probably accompanied by a continuous shrinkage resulting principally from gel formation and that the chemically combined water cannot be evaporated at ordinary temperatures. Of particular importance, however, is the demonstration that, except for the effect of hydration, the gain or loss of colloidal water from the gel is the fundamental cause of volume changes due either to variations in the moisture of the surrounding air or to pressure applied over a period of time.

Although the following explanation is admittedly approximate, it is helpful to visualize the interchange of moisture between the gel and the atmosphere surrounding a mass of concrete, on the basis of a few simple physical principles. The flow of water to or from the gel takes place through minute capillary channels which permeate the mass. The flow along the capillaries, both in direction and amount, is a function of the pressure gradient along these channels. If a moist concrete is placed in dry air, water is evaporated from the outer ends of the capillary channels, thus producing capillary tension within the channels. This tension acts to draw water from the sponge-like clots of gel. The rate of evaporation is, of course, a function of the vapor pressure in the air, so that the drier the air, the more rapid the loss of water from the gel. The gel may be thought of as hygroscopic in nature; the less moisture it contains, the greater its attraction for water and the flatter the moisture gradient along the channels, with a resultant slower rate of moisture loss. The gel nearer the surface of the mass will, of course, dry out more rapidly than that farther from the surface, because of the lesser total frictional resistance to flow along the shorter channels. If the mass is placed either in an atmosphere of 100 per cent relative humidity or under water, there is full vapor pressure on the outside, preventing evaporation, and under this condition the direction of flow is usually inward since the gel will usually take up water. It should be understood that there is always a tendency to establish equilibrium between the outside and inside (vapor) pressures and that in time this may be accomplished.

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[1] C. G. Lynam, "Growth and Movement in Portland Cement Concrete," Oxford University Press, London (1934).

Accompanying changes in moisture content are changes in volume. As moisture is lost, the gel shrinks, which process may be thought of as the collapse of minute pore spaces within the gel; and as moisture is gained, the gel swells as these pore spaces become filled with water.

When an external load is applied to a mass of concrete, the gel is placed under pressure, and moisture is expelled. The rate of expulsion will again be a function of the vapor pressure on the outside of the mass and of the friction in the capillary channels. The greater the load, the steeper the pressure gradient and the more rapid the expulsion of water. This flow of water from the gel due to applied pressure is that which has been defined as seepage. It is clear that by this hypothesis shrinkage due to loss of moisture and time-yield due to seepage are interrelated phenomena, although they may be conveniently considered as separate and additive in their effect.

While it appears that the major portion of the time-yield in concrete is caused by colloidal seepage, nevertheless there is evidence which would indicate that a portion of the total yielding may be due to crystalline or to viscous flow. The flow of rock is observed in nature and it is reasonable to assume that the same phenomena occurs, at least to some extent, in natural aggregates. Considerable differences in time-yield of concrete made of different mineral aggregates seem to substantiate this view. Furthermore it is not impossible for viscous flow to take place in the gel. A finely divided wet clay, which is colloidal in nature, is observed to deform by viscous flow. Glue, a colloid, flows under pressure as does a viscous liquid. The magnitude of lateral flow accompanying the axial flow of concrete lends support to the belief that the yielding may be due, at least in part, to viscous flow.

Scope of the Tests:

The investigations here described were conducted in the Engineering Materials Laboratory of the University of California. The tests were designed to show the effect upon axial flow of concrete under sustained axial compressive stress of the following factors: (1) moisture conditions both as regards storage and as regards the concrete during the period of test, (2) temperature conditions of storage, (3) age at time of loading, (4) magnitude of sustained stress, (5) character and grading of aggregate, (6) size of test specimen, and (7) reinforcement. In addition, tests were made to determine: (1) under conditions of sustained axial loading, the variations in flow between the surface and the center of a mass of concrete and the magnitude of the lateral flow with respect to axial flow, and (2) under triaxial loading conditions, the axial flow when the lateral dimension was maintained constant.

The specimens employed were cylinders varying in diameter from 4 to 18 in. and in length from 8 to 48 in. The reinforced-concrete columns (described later) were 5 in. in diameter and 4 ft. long, having, respectively, 1.9 per cent and 5.0 per cent of longitudinal reinforcement.

The first series of tests was begun in 1926, and the test program has been enlarged from time to time. The number of specimens included in the various series of tests here described is 264. For each group of specimens subjected to sustained loads, there has been maintained a group of unstressed control specimens on which observations were made to determine dimensional changes (such as shrinkage or expansion) due to causes other than stress.

The majority of the test specimens were stored under controlled conditions as regards temperature and humidity. In other cases, the effects of known variations in temperature and humidity have been observed. For air-stored specimens, temperatures varying from 0 to 100 F. (−18 to 38 C.) and relative humidities varying from 25 to 100 per cent have been employed. Water-stored specimens have been maintained at temperatures of 70 and 92 F. (21 and 33 C.).

The ages at time of loading varied from 7 days to 3 months. The sustained loads have been such as to produce concrete stresses as low as 300 lb. per sq. in. and as high as 1200 lb. per sq. in.

The loads, in the majority of the tests, were maintained by steel helical car springs restrained by a rig consisting of rods and plates. The large test specimens were kept under load in a hydraulic testing machine equipped with an automatic load-maintaining apparatus. The triaxial-flow specimens were loaded by hydrostatic pressure acting through copper diaphragms.

Surface measurements of length of specimens were made with strain gages of the fulcrum-plate type, of gage lengths 6, 10 or 20 in., depending upon the size of specimen. Measurements of internal deformations were made with electric strain meters of the resistance-wire type. In the reinforced-concrete column tests, strain-gage measurements were made along gage lines in both the reinforcing steel and the concrete.

Acknowledgments:

The tests here described have been made possible through funds and equipment made available by the University of California and through funds contributed by Engineering Foundation and by the American Society of Civil Engineers through its Committee on Concrete and Reinforced Concrete Arches.

Since the time the tests were begun, more than a score of men have, at one time or another, contributed their services, and thanks are due to them for the painstaking work accomplished. In particular it is desired to acknowledge the assistance of G. E. Troxell, Associate Professor of Civil Engineering, J. L. Banville, Assistant in the Engineering Materials Laboratory, and J. Baines, R. Horonjeff, M. Markoff, J. Rinne, R. Tucker, R. Gauthier, and F. Von der Lage, students in the College of Engineering.

TESTS ON PLAIN CONCRETE

Effect of Moisture Conditions of Storage:

To determine the effect upon flow and shrinkage of various moisture conditions to which a concrete may be exposed, a series of tests was undertaken in which like concrete specimens were stored: (*a*) in air at 50 per cent relative humidity, (*b*) in air at 70 per cent relative humidity, (*c*) in air at 100 per cent relative humidity (fog), and (*d*) under water, all at 70 F. (21 C.).

The results obtained from this series of tests are shown in Fig. 1. Here are plotted, with respect to time, the flow and shrinkage for the concretes under each of the four storage conditions. The upper group of diagrams represents net flow which is calculated to have taken place by subtracting algebraically from the total observed movement (of specimens subjected to the applied load) the observed free change in length of the control specimens, which shrinkage or expansion is given in the lower group of diagrams.

It will be noted in the figure that the flow of concrete stored in air at 70 per cent relative humidity is roughly twice that of the concrete stored at 100 per cent relative humidity while the flow under the condition of 50 per cent relative humidity is 2½ to 3 times that for the moist condition. The flow of the concrete stored in fog and that of the concrete stored under water are practically the same. For the storage at 50 per cent relative humidity the flow over a period of 5½ years amounts to 900 millionths in. per inch which is equivalent to about 1.1 in. per 100 ft., while the total change in length (flow and shrinkage combined) is equivalent to about 2.3 in. per 100 ft. For the given conditions of test, the shrinkage of the concretes stored in air is approximately of the same magnitude as the flow of similarly exposed concretes.

Due to differential shrinkage between the outside and inside of the mass, the control specimens stored at 50 per cent relative humidity developed surface cracks during the period between 400 and 580 days after pressure was applied to the loaded specimens. This is indicated by the dotted line marked *A* in the figure. Since cracking did not occur in the loaded specimens, when the shrinkage correction was applied to the total observed movement, the calculated flow was greater than it normally would have been. The dotted line *B* probably more nearly represents the flow after the cracking of the control specimen.

The effect of the condition of storage with regard to moisture was also determined in a series of tests in which some of the specimens were stored under water and some in air at 70 per cent relative humidity. In this series, various ages at time of loading and various unit stresses were employed. The time-flow and time-shrinkage relations for selected conditions of test are shown in Fig. 2, study of which leads to the following observations. While the flow of the concrete stored in air is always considerably greater than the flow of the corresponding concrete stored under water, the ratios between flows of similar concretes subjected to the two storage conditions is altered by the age at which the sustained load is first applied. For example, for concrete loaded at the age of 28 days to 900 lb. per sq. in. the flow for the dry storage condition is twice that for the water storage condition, while for the concrete loaded at 3 months to 1200 lb. per

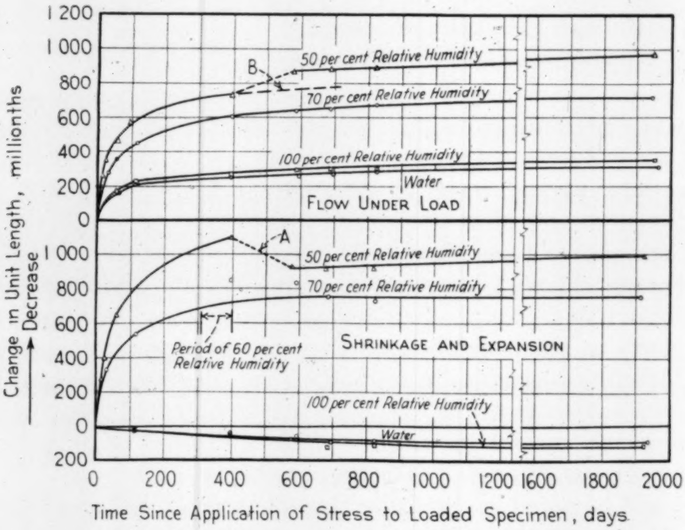

FIG. 1.—Effect of Moisture Condition of Storage upon Flow and Shrinkage.

Flow tests, series No. 3. Specimens, 4 by 14-in. cylinders. Aggregate, gravel. Fineness modulus = 5.61. Cement, normal portland. Aggregate-cement ratio = 5.67 by weight. Water-cement ratio = 0.89 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Sustained stress 800 lb. per sq. in. Storage after loading at 70 F. (21 C.), humidity as indicated.

sq. in. the flow for the dry storage condition is generally nearly 3 times that for water storage. It is apparent that the extent to which hydration has proceeded before load is applied has a marked effect upon the flow.

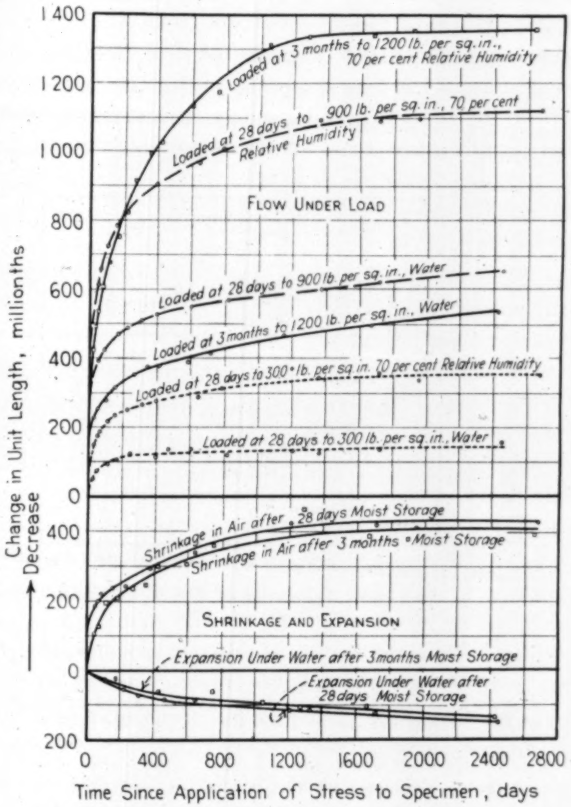

FIG. 2.—Effect of Moisture Condition of Storage upon Flow and Shrinkage.

Flow tests, series No. 2. Specimens, 4 by 14-in. cylinders. Aggregate, crushed granite. Fineness modulus = 5.03. Cement, normal portland. Aggregate-cement ratio = 5.05 by weight. Water-cement ratio = 1.03 by volume. Curing, moist at 70 F. (21 C.) until time of loading. Age at loading, as indicated. Sustained stress, as indicated. Storage after loading, in air at 70 F. (21 C.), 70 per cent relative humidity, or water at 70 F. (21 C.), as indicated.

For the concrete stored in air there appears to be a marked decrease in the rate of flow at the later ages, while this does not appear to be so marked for the concrete stored under water. Perhaps the most striking feature indicated by these data is the fact that, after 7 years of sustained stress, for certain conditions some flow is still taking place.

The flow of the concrete loaded at the age of 3 months to a stress of 1200 lb. per sq. in. amounts after a period of 7 years to a deformation equivalent to about $1\frac{1}{2}$ in. per 100 ft., and the flow and shrinkage combined result in a change in length equivalent to more than 2 in. per 100 ft. during this period of time. It should be observed, however, that in moist-stored concrete, the free expansion is in a direction opposite to the direction of flow, so that the total deformation is less than the flow alone.

In each of the tests described above, the concretes stored under each moisture condition were as nearly as possible the same as regards composition, size of mass, curing, and sustained stress. These data appear to substantiate the view, discussed in the introduction, that the seepage of water from the concrete mass results in a continued yielding of the mass. The magnitudes of flow obtained indicate that the seepage is a large contributing factor to time-yield.

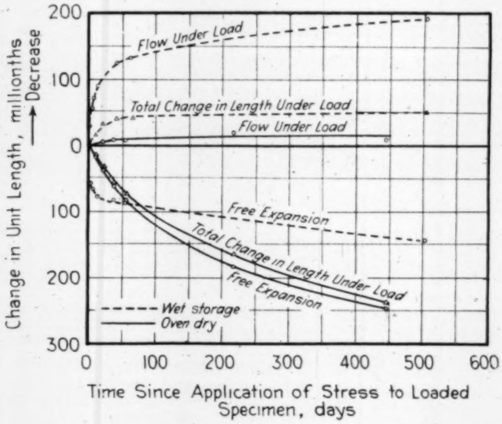

FIG. 3.—Effect of Moisture Content of Concrete upon Time-Deformations.

Flow tests, series No. 14. Specimens, 6 by 12-in. cylinders. Aggregate, gravel. Fineness modulus = 5.69. Cement, normal portland. Aggregate-cement ratio = 6.95 by weight. Water-cement ratio = 0.92 by volume. Curing, moist at 70 F. (21 C.), for 3 months. Age at loading, 3 months. Sustained stress, 800 lb. per sq. in. Preparation for test, dry specimen oven-dried to constant weight for 5 days before test, then covered with 3 coats of lacquer. Storage after loading, oven-dried specimens, in air at 70 F. (21 C.), 50 per cent relative humidity; wet specimens, water at 70 F. (21 C.).

Effect of Moisture Content of the Concrete:

To determine whether or not flow would take place in a perfectly dry concrete, a series of tests was undertaken in which one group of specimens was oven-dried to constant weight before load was applied, while a companion group, after loading, was stored under water.

The concrete was moist-cured for three months before the tests were begun, in order to permit the hydration of the cement to take place to a considerable degree. The oven-dried specimens were covered with three coats of lacquer to reduce the absorption of moisture from the air by the concrete during the period of test.

The results of these tests are shown in Fig. 3 in which there is plotted with respect to time, for both the oven-dried and the water-stored specimens, the total changes in length as well as the calculated flows. The behavior of the oven-dried group is of particular interest. Regardless of the fact that the concrete is stored in air at 50 per cent relative humidity and that a lacquer coating is used, an appreciable amount of water is absorbed, resulting in an expansion during the first year of about 250 millionths. The attraction of the dried gel for water must have been very great, for in spite of the sustained stress of 800 lb. per sq. in. the total change in length (expansion) of the concrete under load over the same period is about 240 millionths. These facts are significant in that they give insight into the relation of moisture and pressure to volume change. Evidently the applied stress, instead of expelling water of which there was none in excess, restrained the gel from taking up as much water as could be taken up under conditions of no stress. The calculated flow, although very small, represents not an actual decrease in length but a measure of the restraint to the increase in length. The relative amounts of the flow and shrinkage should also serve to indicate something of the magnitude of the forces that must exist to bring the gel into a condition of equilibrium as regards moisture content.

The behavior of the water-stored specimens illustrates the same relation, but to a different degree. The water-stored concrete under no load expands, but under stress not only is expansion prevented but also a decrease in length occurs with time. The stress evidently has been great enough to expel some water from the gel, even though the gel was capable of taking up water under conditions of no restraint. It is also of interest that the water-stored control specimens evidently will have reached equilibrium saturation while the corresponding oven-dried group is still taking up water.

Effect of Temperature of Storage:

To study the effect of temperature of storage upon flow, a series of tests was undertaken in which groups of specimens were stored in air at 0, 70, and 100 F. (−18, 21, and 38 C.), and under water at 70 and 92 F. (21 and 33 C.). The relative humidity in the 70 F. (21 C.) storage room was 50 per cent and that for the 100 F. (38 C.) storage room was 25 per cent. The specimens were brought to constant temperature at each of the various storage conditions before the loading tests were begun.

The results of the tests, given in Fig. 4, indicate the flow and shrinkage obtained over a period of time for concrete at each of the given temperatures. It is important to note that for the condition of water storage, the flow of the concrete stored at 92 F. (33 C.) is but a very small amount greater than the flow of the concrete stored at 70 F. (21 C.). This would indicate that if the moisture conditions are the same, the effect upon flow of temperature variation within the range studied is at least slight and is perhaps negligible. The flow of the concrete stored in air at 0 F. (−18 C.) is of the same order of magnitude as that for the water-storage condition at the higher temperatures. It is not unlikely that the low rate of flow is due, at least in part, to a very low rate of evaporation of moisture at the freezing temperature.

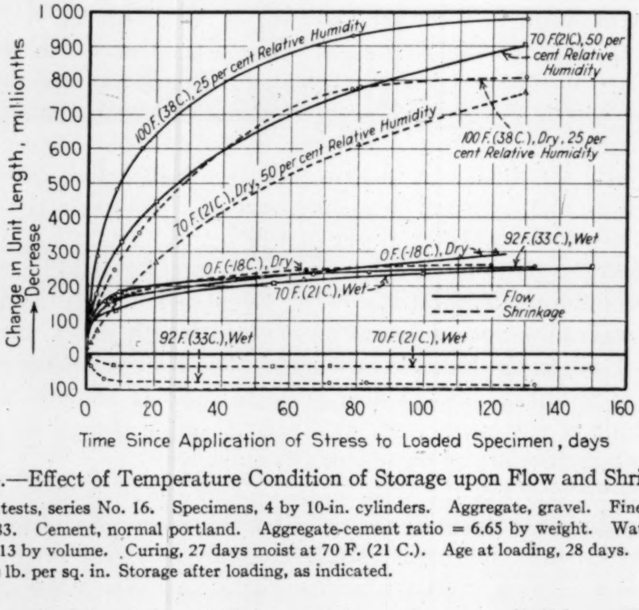

FIG. 4.—Effect of Temperature Condition of Storage upon Flow and Shrinkage.

Flow tests, series No. 16. Specimens, 4 by 10-in. cylinders. Aggregate, gravel. Fineness modulus = 4.83. Cement, normal portland. Aggregate-cement ratio = 6.65 by weight. Water-cement ratio = 1.13 by volume. Curing, 27 days moist at 70 F. (21 C.). Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. Storage after loading, as indicated.

While for the concrete stored in air the greatest flow is obtained for the highest temperature condition, this is undoubtedly due to the lower humidity of the air at the higher temperatures. The similarity between the rates of shrinkage and flow under each of the dry temperature conditions appears to confirm this. It is also noteworthy that the magnitude of flow for the 70 F. (21 C.) storage is approaching that for the 100 F. (38 C.) storage, a condition which may be partly attributable to the relative rates of hydration of the cement over the period of test, as well as to the relative rates of evaporation of moisture.

This series of tests serves further to substantiate the view that seepage is the major cause of volume changes due to stress.

Effect of Grading and Character of Aggregate:

To study the influence of the aggregate upon flow and shrinkage, two series of tests were made for which the variables were (*a*) the grading of the coarse aggregate and (*b*) the mineral character of the aggregate.

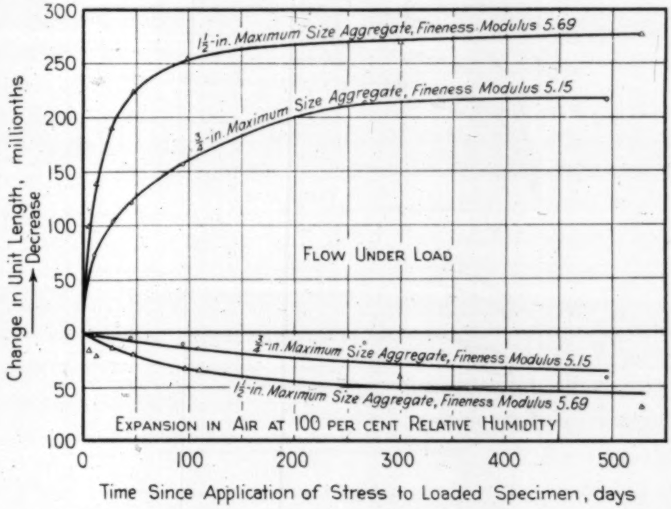

FIG. 5.—Effect of Gradation of Coarse Aggregate upon Flow and Expansion.

Flow tests, series No. 13. Specimens, 6 by 12-in. cylinders. Aggregate, gravel. Fineness modulus as indicated; the finer mix was obtained by substituting for the $\frac{3}{4}$ to $1\frac{1}{2}$-in. aggregate, used in the coarser mix, an equal solid volume of $\frac{1}{4}$ to $\frac{3}{4}$-in. aggregate. Cement, normal portland. Aggregate-cement ratio = 6.95 by weight. Water-cement ratio = 0.92 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. Storage after loading, fog (100 per cent relative humidity), 70 F. (21 C.).

In the tests to determine the effect of grading, two similar groups of specimens were prepared, alike in aggregate-cement ratio and water-cement ratio, but differing in the maximum size of coarse aggregate. A difference in consistency of the fresh concrete, of course, was obtained. For one group of specimens the maximum size of aggregate was $1\frac{1}{2}$ in. and for the other the maximum size was $\frac{3}{4}$ in.

In Fig. 5 are plotted, with respect to time, the flow and shrinkage in each of the two concretes. It was found that the flow of the concrete having the finer gradation of aggregate was roughly between one-half and three-fourths of the flow of the concrete having the coarser gradation of aggregate. It is possible that this difference in flow is due, in part, to the difference in the total surface areas of the aggregate particles in the two concretes. The concrete having the finer gradation of aggregate has a thinner film of cement gel around each aggregate particle, and offers a greater frictional resistance to the relative movements of particles within the mass. The free expansion, under moist storage conditions, of the concrete of finer gradation was on the average one-half to two-thirds of the expansion of the concrete of the coarser gradation. Thus the data indicate considerable relative differences in volume changes as the result of a change in grading of aggregate, which aggregate is of the same general character throughout its range of size, although for the given conditions of test the absolute difference between total movements of the two concretes is not large.

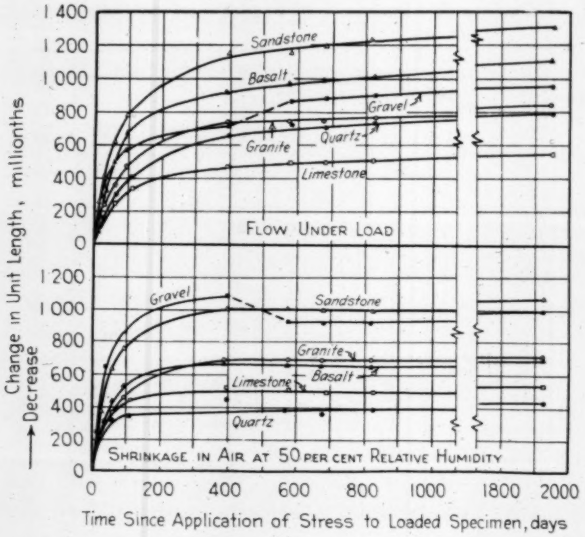

FIG. 6.—Effect of Mineral Character of Aggregate upon Flow and Shrinkage.

Flow tests, series No. 4. Specimens, 4 by 14-in. cylinders. Aggregate, as indicated. Fineness modulus = 5.75 except gravel which had fineness modulus = 5.61. Cement, normal portland. Aggregate-cement ratio = 5.67 by weight. Water-cement ratio = 0.89 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. Storage after loading, air at 70 F. (21 C.), 50 per cent relative humidity.

To determine the effect upon flow and shrinkage of the mineral character of the aggregate, a series of tests was made in which each of six groups of specimens contained a different aggregate. The aggregates included a local gravel, sandstone, basalt, granite, quartz, and limestone. Within each group, the same mineral aggregate was used from fine to coarse; the grading was the same for all groups.

The results of the tests are shown in Fig. 6, in which are plotted, with respect to time, the flow and the shrinkage of the concretes containing each of the different aggregates. It will be noted that after a period of about 5 years the maximum flow (1300 millionths) is exhibited by the sandstone concrete and the minimum (550 millionths) by the limestone. The quartz and granite whose mineral compositions are somewhat similar exhibit approximately the same deformation under load. The range in flow is more than 100 per cent of the magnitude of flow of the limestone concrete.

While in general the concrete which exhibits a large flow also shows a high shrinkage, if the individual aggregates are considered it is seen that the magnitude of shrinkage is not a criterion of the magnitude of flow. It is of interest to note that the shrinkage of the quartz concrete is roughly one-half of that of the granite concrete. The gravel concrete gave the highest measured shrinkage until surface cracks developed at the age of about 400 days, as discussed in connection with Fig. 1. The flow of the gravel concrete, however, is between the extreme ranges in flow observed for the other concretes.

It seems reasonable to believe that the differences in flow are due to causes other than seepage. The fact that the aggregates were batched in a saturated, surface-dry condition, and the fact that for most of the aggregates used the absorption factors were low, seems to preclude the possibility of marked variations in the results due to differences in moisture conditions of the aggregates themselves. As yielding takes place within a mass of concrete, there must occur relatively high concentrations of stress on some of the aggregate particles, such that slip or crystalline flow may take place. Differences in stress distribution among the particles due to differences in modulus of elasticity of the various kinds of aggregate, as well as differences in crystalline behavior between the various mineralogical structures, may account for the differences in flow.

Effect of Size of Mass:

To determine the effect upon flow of size of mass, a series of tests was undertaken for which the specimens were cylinders 6, 8 and 10 in. in diameter, all subjected to the same intensity of stress. To eliminate the effect of varying internal moisture conditions, all the specimens were stored in fog at 100 per cent relative humidity.

The results of the tests are shown in Fig. 7. It is seen that the larger the specimen the smaller the flow obtained in a given period of time. The flow of the 10-in. specimens at the end of 500 days is about one-half that of the 6-in. specimens, while the flow of the 8-in. specimens is intermediate between that of the larger and smaller specimens. The differences in flow between the various sizes of concrete masses are undoubtedly due largely to differences in the rate of seepage. In the larger masses, the total length of channel through which water must pass when expelled by pressure is greater, with a resulting greater total frictional resistance to the flow of the water.

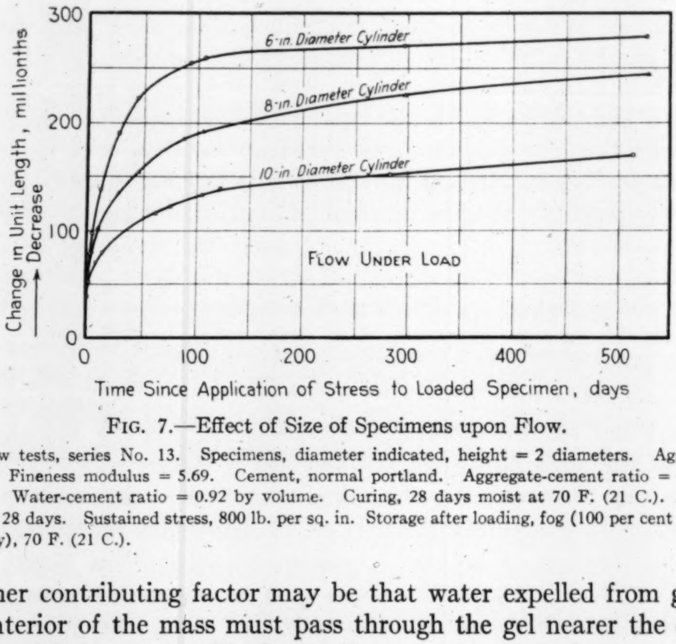

FIG. 7.—Effect of Size of Specimens upon Flow.

Flow tests, series No. 13. Specimens, diameter indicated, height = 2 diameters. Aggregate, gravel. Fineness modulus = 5.69. Cement, normal portland. Aggregate-cement ratio = 6.95 by weight. Water-cement ratio = 0.92 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. Storage after loading, fog (100 per cent relative humidity), 70 F. (21 C.).

Another contributing factor may be that water expelled from gel in the interior of the mass must pass through the gel nearer the outer portions of the mass. The larger the concrete mass, the greater the supply of water that can be drawn from the interior, with the result that the gel in the outer parts of the mass is less readily consolidated.

Variations in Flow Within a Mass—Lateral Flow:

Since there must normally exist considerable variations in moisture content within a mass of concrete exposed to dry air, it is reasonable to suppose that the rate and magnitude of flow and shrinkage would not be uniform throughout the mass. Tests to study these variations were made on a pair of 18 by 36-in. cylinders, stored in the air of the laboratory. Measurements of deformation were made along surface gage lines and along the axis of each specimen. Measurements of internal deformation were also made along two mutually perpendicular diameters near the mid-section of the specimens; these measurements afforded an indication of the extent of lateral flow under load. Since these tests were by no means exhaustive and since they extended over a period of but 25 days, the results are to be regarded only as indicative of probable trends.

In Fig. 8 are plotted the flow of the loaded specimen and the free change in length of the control specimen. The longitudinal flow at the surface of the loaded specimen is considerably greater than the flow at the center. This may be attributed to the difference in the ease with which the moisture is lost from the parts of the mass. This is also indicated by the behavior of the control specimen in which the surface tended to shrink while the central portion remained practically stationary. Of importance is the observed lateral flow, which is approximately one-fifth of the (internal) longitudinal flow. This fact would seem to indicate that viscous or crystalline flow, as well as yielding due to seepage, is taking place. These findings with respect to lateral flow are contrary to the tentative findings of a previously

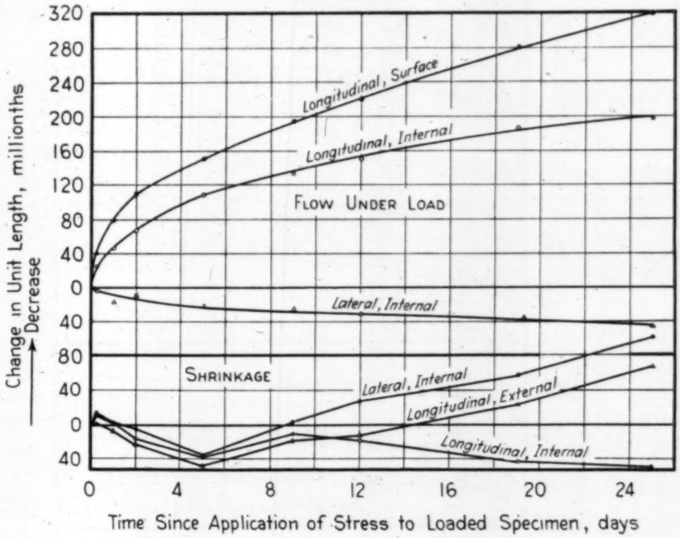

Fig. 8.—Axial and Lateral Flow in 18 by 36-in. Cylinders.

Flow tests, series No. 15. Specimens, 18 by 36-in. cylinders. Aggregate, gravel. Fineness modulus = 5.69. Cement, normal portland. Aggregate-cement ratio = 6.95 by weight. Water-cement ratio = 0.92 by volume. Curing, 2 months moist at 70 F. (21 C.). Age at loading, 2 months. Sustained stress, 1200 lb. per sq. in. Storage after loading, air of laboratory.

reported[1] series of tests in which no appreciable lateral flow was observed over a short period of time. It is believed that the test results here reported more nearly represent the true condition due to (1) the use of measuring instruments of greater precision, (2) the use of a higher sustained stress, and (3) the fact that the measurements were made within the mass, with a consequent reduction of the effect of shrinkage.

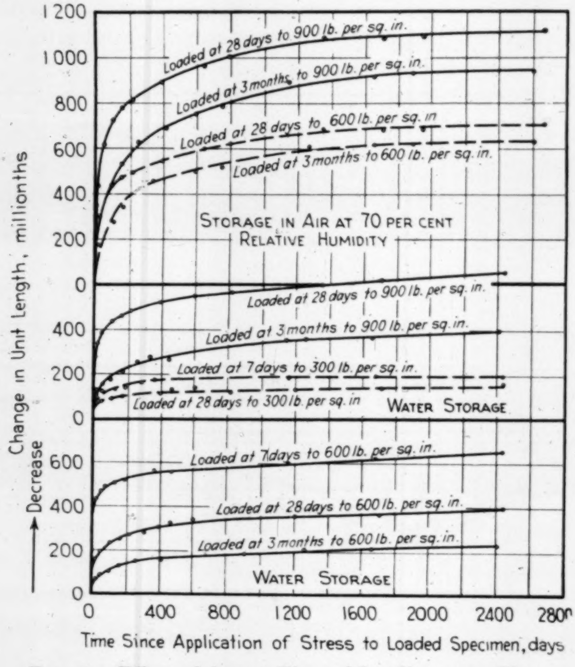

FIG. 9.—Effect of Age at Time of Loading upon Flow.

Flow tests, series No. 2. Specimens, 4 by 14-in. cylinders. Aggregate, granite. Fineness modulus = 5.03. Cement, normal portland. Aggregate-cement ratio = 5.05 by weight. Water-cement ratio = 1.03 by volume. Curing, moist at 70 F. (21 C.) until time of loading. Age at loading, as indicated. Sustained stress, as indicated. Storage after loading, air at 70 F. (21 C.), 70 per cent relative humidity or water at 70 F. (21 C.), as indicated.

Effect of Age at Time of Loading:

The effect of age at time of loading was determined by a series of tests in which specimens of the same concrete mixes were loaded to various intensities of stress at ages of 7 days, 28 days, and 3 months. The results of the tests are shown in Fig. 9. In the lower group of diagrams is shown the flow obtained over a period of 2400 days for concretes stored under water and stressed to 600 lb. per sq. in. It will be noted that the flow of the concrete loaded at the age of 28 days is roughly twice that of the concrete loaded at 3 months, while the flow of the concrete loaded at 7 days is approximately three times that of the concrete loaded at 3 months. It should also be noted that the rate of flow during the first few weeks under load is much greater for the concretes loaded at the earlier ages than for the concretes loaded at the later ages. The same general observations apply to the middle group of diagrams, which represent the flows for concretes stored under water but stressed to 300 and 900 lb. per sq. in.

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[1] R. E. Davis and H. E. Davis, "Flow of Concrete Under the Action of Sustained Loads," *Proceedings*, Am. Concrete Inst., Vol. 27, p. 871 (1931).

TABLE I.—EFFECT UPON FLOW OF INTENSITY OF SUSTAINED STRESS.

Flow Tests, Series No. 2. Specimens, 4 by 14-in. cylinders. Aggregate, granite. Cement, normal portland. Aggregate-cement ratio = 5.05 by weight. Water-cement ratio = 1.03 by volume. Fineness modulus = 5.03. Curing, moist at 70 F. (21 C.) to time of loading. Age at time of loading, as indicated. Storage after time of loading, at 70 F. (21 C.) as indicated.

Storage	Age at Time of Loading, days	Period of Sustained Stress, years	Flow Under Load, millionths			
			Stress, 300 lb. per sq. in.	Stress, 600 lb. per sq. in.	Stress, 900 lb. per sq. in.	Stress, 1200 lb. per sq. in.
Air of 70 per cent relative humidity	28	1	260	550	900	...
		6½	350	700	1110	...
	90	1	...	450	690	990
		6½	...	630	940	1360
Water	7	1	170	550	...	...
		6½	190	650	...	...
	28	1	120	300	520	...
		6½	150	390	650	...
	90	1	...	160	270	370
		6½	...	220	390	530

In the upper group of diagrams is shown the flow obtained for concretes in dry storage. Under this condition the effect of age at time of loading is not nearly so marked as for the water-storage condition. Here, for either the 600 or 900 lb. per sq. in. stress, the flow of the concretes loaded at 28 days is only of the order of 10 to 20 per cent greater than the flow of the concretes loaded at 3 months.

A study of the figure as a whole indicates that for a given intensity of sustained stress and a given storage condition, the *rate* of flow after a year or so under load is practically independent of the age at time of loading. Further, the difference between the flows of concretes loaded at the various ages is more marked for the higher sustained stresses than for the lower stresses.

These tests indicate the effect upon flow of the extent to which hydration of the cement and hardening of the gel has taken place.

Effect of Intensity of Sustained Stress:

The effect upon flow of intensity of sustained stress was also obtained in the test series described in the previous section. Within the various groups of specimens, stresses of 300, 600, 900, and 1200 lb. per sq. in. were employed. In Table I are tabulated for the various test conditions the flows resulting from the given stresses over periods of 1 year and 6½ years.

The concretes loaded at ages of 28 and 90 days exhibit at the ends of the given periods of sustained stress, flows which may be said to be roughly proportional to the applied stresses. However, there are deviations from this relation. The flow appears to be relatively greater in proportion to intensity of stress at the higher sustained stresses than at the lower stress. This is particularly evident in the wet-stored concrete, and applies also to the concretes loaded at the age of 7 days.

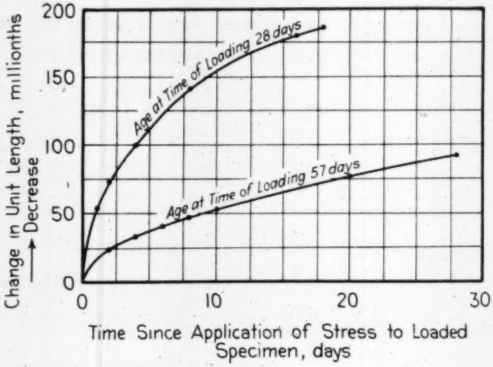

FIG. 10.—Axial Flow Under Constant Axial Load and Constant Lateral Dimensions.

Flow tests, series No. 12. Specimens, 12 by 48-in. cylinders. Aggregate, gravel. Fineness modulus = 5.69. Cement, normal portland. Aggregate-cement ratio = 6.95 by weight. Water-cement ratio = 0.93 by volume. Curing, in sealed copper containers at 70 F. (21 C.). Age at loading, as indicated. Sustained axial stress, 1090 lb. per sq. in. Lateral (radial) pressures to maintain constant lateral dimensions: Specimen No. 1—210 to 155 lb. per sq. in. Specimen No. 2—278 to 183 lb. per sq. in. Storage after loading, in sealed copper containers at 70 F. (21 C.).

While a straight-line stress-flow relationship would be convenient in estimating the probable magnitude of stress or deformation in a concrete structure, it is believed that the use of such a relation should be considered with caution.

Flow Under Triaxial Loading:

A brief study of flow under triaxial loading was made in which there was investigated the effect of maintaining constant axial load and constant lateral dimension over a period of time. The loads were applied by means of hydraulic pressure acting through thin copper diaphragms. The lateral pressures were applied radially against the circumferential surface of the cylinders. The strains were measured by strain meters embedded in the concrete. The specimens were kept in sealed containers to prevent loss of moisture; under such conditions, the shrinkage may be taken as practically negligible.

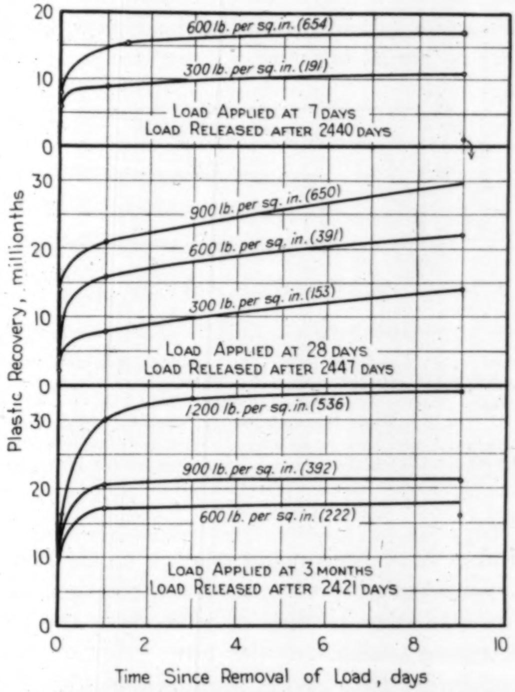

FIG. 11.—Plastic Recovery After 6¾ Years of Sustained Load.

Flow tests, series No. 2. Specimens, 4 by 14-in. cylinders. Aggregate, granite. Fineness modulus = 5.03. Cement, normal portland. Aggregate-cement ratio = 5.05 by weight. Water-cement ratio = 1.03 by volume. Curing, moist at 70 F. (21 C.) until time of loading. Age at loading, as indicated. Age at release of load, as indicated. Previously sustained stress, indicated on each curve. Plastic flow in millionths before release of load, indicated on each curve in parenthesis. Storage before and after release of load, water at 70 F. (21 C.).

The deformations, with respect to time, under load are shown in Fig. 10, in which it is seen that the concrete loaded at the age of 28 days flows about twice as much as the concrete loaded at 57 days. It should be noted that for the condition of lateral restraint, the deformations with respect to time represent, to some scale, a decrease in volume.

It was found that the lateral pressures required to maintain constant lateral dimension decreased rapidly in the first day or two after loading, and more slowly at the later ages. The radial pressures for the specimen loaded at 28 days varied over the period of test from 278 to 183 lb. per sq. in., and those for the specimen loaded at 57 days varied from 210 to 155 lb. per sq. in.

Perhaps the most significant result of these tests is that the axial flow occurring when the lateral dimension is maintained constant is of the same order of magnitude as the flow observed in similar concretes under no lateral restraint.

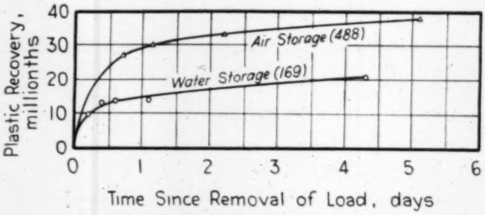

FIG. 12.—Plastic Recovery After 11 Months Under Load.

Flow tests, series No. 7. Specimens, 6 by 12-in. cylinders. Aggregate, gravel. Fineness modulus = 5.70. Cement, normal portland. Aggregate-cement ratio = 4.00 by weight. Water-cement ratio = 0.63 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Age at release of load, 1 year. Previously sustained stress, 800 lb. per sq. in. Plastic flow in millionths before release of load, indicated on each curve in parenthesis. Storage before and after release of load, in air at 70 F. (21 C.), 50 per cent relative humidity, or in water at 70 F. (21 C.) as indicated.

Plastic Recovery:

Some studies have been made relative to the change in length of concrete, over a period of time, after the release of sustained load. Because of its similarity to flow or time-yield, the phenomenon is called *plastic recovery*, although the term "*elastic after-effect*," used by some investigators, may also be appropriate.

In Fig. 11 is shown the effect of magnitude of previously sustained stress upon the plastic recovery of specimens loaded at various ages and stored under water. These specimens have been under load for more than 6½ years, yet an appreciable recovery is observed. Some recovery is still taking place 9 days after the loads are released. It will be noted that for any given age at loading the higher the previously sustained stress or the higher the magnitude of flow before release of load (shown in parenthesis on the curves), the greater the recovery.

The results of observations to determine the plastic recovery of two groups of specimens after 11 months under stress of 800 lb. per sq. in. are shown in Fig. 12. The specimens which were air-stored exhibit about twice the recovery of the specimens stored under water. The rate of recovery is still appreciable five days after the load is released.

Observations to determine plastic recovery were also made in a series of tests on wet-stored and dry-stored concretes to which stresses of 800 lb. per sq. in. and 40 lb. per sq. in. had been applied alternately for one-day intervals over a period of 30 days. The average stress over the whole period was 420 lb. per sq. in. and the residual deformations resulting from flow were 245 and 125 millionths for the wet- and dry-stored concretes, respectively.

In Fig. 13 are plotted the amounts of recovery obtained over a period of 200 days. The dry-stored concrete exhibits a greater recovery than the wet-stored concrete, the relative amounts of recovery being approximately in the ratio of the flows previously obtained, or about 2:1. The recovery is apparently complete within about two months after release of load.

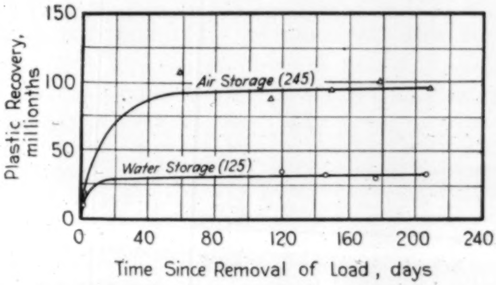

FIG. 13.—Plastic Recovery After 1 Month Under Alternating Loads.

Flow tests, series No. 6. Specimens, 4 by 14-in. cylinders. Aggregate, gravel. Fineness modulus = 4.86. Cement, normal portland. Aggregate-cement ratio = 5.68 by weight. Water-cement ratio = 0.85 by volume. Curing, 28 days moist at 70 F. (21 C.). Age at loading, 28 days. Age at total release of load, 58 days. Previously sustained load, 800 lb. per sq. in. for 1 day, 40 lb. per sq. in. for 1 day, alternating for period of 30 days. Average stress 420 lb. per sq. in. Residual (plastic) deformation in millionths before total release of load, indicated on each curve in parenthesis. Storage before and after release of load, air at 70 F. (21 C.) and 50 per cent relative humidity or water at 70 F. (21 C.), as indicated.

A reasonable explanation of this phenomenon of plastic recovery is as follows: the aggregate particles having been deformed elastically by application of load are restrained from full recovery of the original volume, upon release of load, by the film of cement gel which has hardened and which has been consolidated during the period of sustained stress. For some time after release of load the gel is in tension and a "reverse seepage"[1] takes place. The fact that the relative magnitudes of recovery observed are approximately in the same order as the relative plastic flows under the previously sustained loads seems to substantiate this view.

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[1] C. G. Lynam, "Growth and Movement in Portland Cement Concrete," Oxford University Press, London (1934).

TESTS ON REINFORCED CONCRETE COLUMNS

To determine the variation in stress, due to plastic flow and shrinkage, in reinforced concrete columns, a series of tests was undertaken in which concretes of two different strengths and two steel ratios were employed. For one group of tests the ends of the reinforcing bars were flush with the ends of the columns, so that a portion of the load was applied directly to the steel. For the other groups, the steel was recessed, extending only to within 2 in. of the ends of the columns, so that the load carried by the steel was transferred to it through bond with the concrete. The flow and shrinkage of plain concretes corresponding to those used in the reinforcing columns were determined from an auxiliary group of specimens.

TABLE II.—CONCRETE MIXES USED IN REINFORCED CONCRETE COLUMN TESTS.

Aggregate, local sand and gravel, 0 to $\frac{1}{2}$ in. Cement, normal portland. Consistency, 6-in. slump.

Compressive Strength at 28 Days, lb. per sq. in.		Secant Modulus of Elasticity at 28 Days (at stress of 800 lb. per sq. in.), lb. per sq. in.	Aggregate-Cement Ratio, by weight	Water-Cement Ratio, by volume
Nominal	By Test			
2000	1730	2 800 000	8.40	1.24
4000	3780	4 000 000	4.80	0.80

The reinforced columns were 5 in. in diameter and 48 in. long, reinforced longitudinally with square deformed bars and laterally with wire spiral hooping having an outside diameter of 5 in. The plain concrete specimens were 5 by 14-in. cylinders. With concrete designed for a strength of 2000 lb. per sq. in., steel ratios of 1.9 and 5.0 per cent were employed; with concrete designed for a strength of 4000 lb. per sq. in., a steel ratio of 1.9 per cent was employed. In the columns having the longitudinal steel ratio of 1.9 per cent, the spiral steel ratio was 0.7 per cent; with the longitudinal steel ratio of 5.0 per cent, a spiral steel ratio of 1.8 per cent was used. The characteristics of the concrete mixes are shown in Table II. The steel used for the longitudinal reinforcing was found to have a yield strength of 42,500 lb. per sq. in., a tensile strength of 62,400 lb. per sq. in., and a modulus of elasticity of 29,500,000 lb. per sq. in.

After 21 days of moist-curing at 70 F. (21 C.), half of the specimens in each test group were stored under water at 70 F. (21 C.)

and the other half were stored in air of 50 per cent relative humidity at 70 F. (21 C.). Load was applied at the age of 28 days.

The column loads were determined in accordance with the provisions of the 1924 Report of the Joint Committee on Standard Specifications for Concrete and Reinforced Concrete.[1] Loads were applied to the plain concrete cylinders such that the stresses would correspond to those at time of loading in the columns having the 1.9 per cent steel ratio. The stresses in the reinforced column due to application of load are given in Table III.

Stresses Developed in Reinforcing Steel Due to Sustained Load and Shrinkage:

In Fig. 14 are shown the variations in stress in the reinforcing steel, over the period of test, for columns in which the ends of the steel bars were flush with the ends of the columns. Figure 14 (*a*) gives the stresses for columns stored in air and Fig. 14 (*b*) gives the stresses for columns stored under water. The stresses indicated have been calculated from strain observations on the steel, which observations were begun at the age of 2 days.

TABLE III.—STRESSES IN REINFORCED COLUMNS DUE TO APPLICATION OF LOAD.

Loads from which stresses were calculated were determined according to provisions of the 1924 Report of the Joint Committee on Specifications for Concrete and Reinforced Concrete.

Concrete Strength, lb. per sq. in.	Steel Ratio, per cent	Steel Stress, lb. per sq. in.		Concrete Stress, lb. per sq. in.	
		Air Storage	Water Storage	Air Storage	Water Storage
2000	1.9	6540	5460	612	606
	5.0	9660	7200	875	810
4000	1.9	7860	7320	976	927

In each section of the figure, the lower group of diagrams represents the stresses induced in the steel by concrete shrinkage or expansion alone. The upper group of diagrams in each figure represents, for the columns under load, the total stresses resulting: (*a*) from shrinkage or expansion, (*b*) from instantaneous deformation due to applied load, and (*c*) from plastic flow due to continuous application of the load.

Examination of the figures shows that for columns stored in air, the steel stress due to shrinkage in the 4000-lb. (richer) concrete is more than twice that of the 2000-lb. (leaner) concrete; the stress after the age of 1000 days is about 15,000 lb. per sq. in. for the former as against 6000 lb. per sq. in. for the latter. Also, the use of 5.0 per cent instead of 1.9 per cent of longitudinal steel results in a reduction of about 50 per cent in the stress due to shrinkage. For non-loaded columns stored in water the stresses are practically negligible.

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[1] *Proceedings,* Am. Soc. Testing Mats., Vol. 24, Part I, p. 303 (1924).

In comparing the relative deformations and stresses of the columns under load, it should be remembered that, due to the method of selecting the allowable load, the initial stresses in the concrete are not the same. It will be noted that after a period of 1000 days, under the loads permitted by the specifications, the steel stress in columns having 2000-lb. concrete and 5.0 per cent steel is only 27,000 lb. per sq. in., while in the column having the 4000-lb. concrete and 1.9 per cent steel the steel stress is 40,000 lb. per sq. in. Due to the smaller load, as well as to the lesser shrinkage, the steel stresses in the columns made of the 2000-lb. concrete are somewhat less than those in the corresponding columns made of the 4000-lb. concrete.

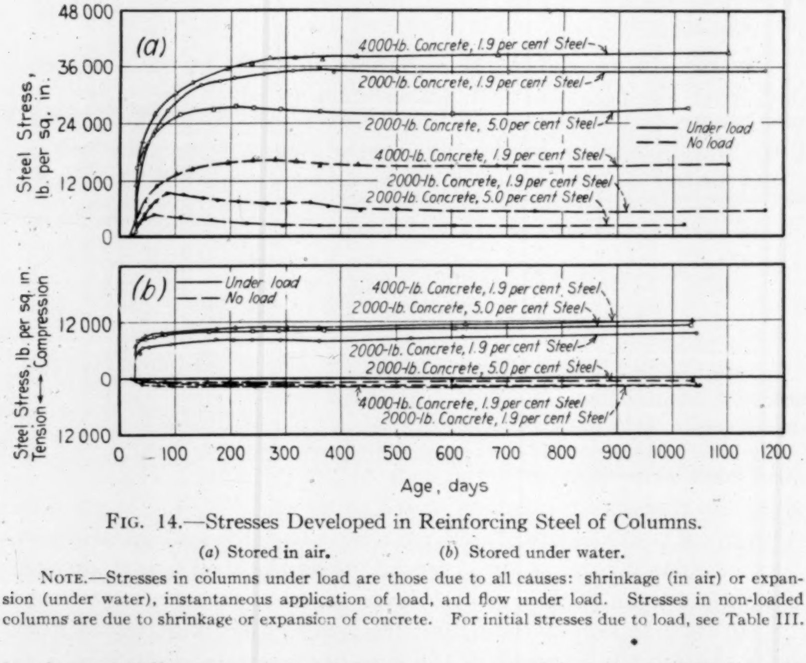

FIG. 14.—Stresses Developed in Reinforcing Steel of Columns.

(a) Stored in air. (b) Stored under water.

NOTE.—Stresses in columns under load are those due to all causes: shrinkage (in air) or expansion (under water), instantaneous application of load, and flow under load. Stresses in non-loaded columns are due to shrinkage or expansion of concrete. For initial stresses due to load, see Table III.

In the columns stored in air the *change* in stress is practically zero after about one year, while in the columns stored under water equilibrium is reached in about two months. The steel stresses in the loaded columns stored under water are generally about one-third to one-half the stresses in the columns stored in air.

It is noteworthy that over the period of observation the maximum steel stress developed (about 40,000 lb. per sq. in.) is but little less than the yield strength of the reinforcing steel (42,500 lb. per sq. in.).

Variations in Concrete Stresses Due to Applied Load and to Shrinkage or Expansion:

The variation in average concrete stresses with respect to time, for the columns in which the steel was flush with the ends, are shown in Figs. 15 and 16. These stresses at given ages were calculated from the difference between the total applied loads and the loads carried by the reinforcing steel.

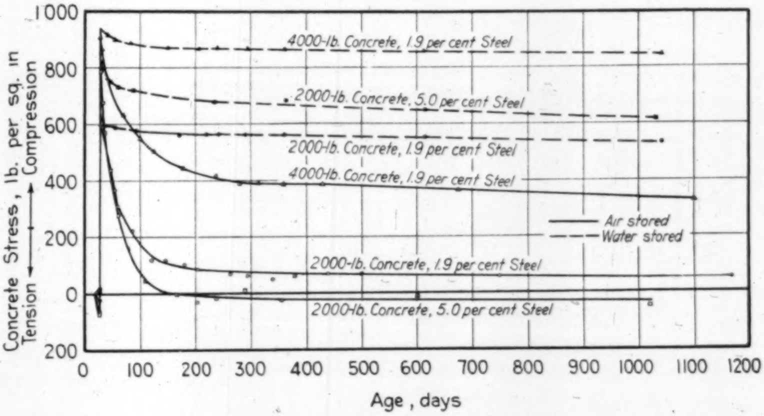

FIG. 15.—Variations in Concrete Stresses in Reinforced Columns Under Load.

NOTE.—The concrete stresses are calculated *average* stresses over the cross-section of the columns obtained from the difference between the total applied load and the total steel stress determined by strain observations. For initial stresses due to load, see Table III.

The stresses in the concrete of the column under load are shown in Fig. 15. After the application of load the compressive stresses decrease at first rapidly and then more slowly, until a condition of equilibrium is reached. Due to the greater rate at which the air-stored columns deform, the reduction of concrete stress is much more rapid than in the case of the water-stored columns. In the air-stored columns made of the 2000-lb. concrete and having the 5.0 per cent steel ratio, the steel is calculated to be carrying all the load after about 7 months, while the reduction in concrete compressive stress in the columns having 4000-lb. concrete and a 1.9 per cent steel ratio is, after a year under load, about 50 per cent. The reductions in concrete stress in the water-stored columns over the period of observation are of the order of 10 to 25 per cent.

In Fig. 16 are shown the concrete stresses calculated to exist in the columns under no load. Here, due to shrinkage, tensile stresses are developed in the air-stored columns of the order of 100 to 300 lb. per sq. in. while, due to expansion, compressive stresses of less than 50 lb. per sq. in. are developed in the water-stored columns. After one to two months in air, the concrete in the columns having the leaner mix reaches its tensile strength, and with the development of cracks, the stress is relieved to a considerable extent. The calculated *average* stress at which cracking occurs in these columns is about 200 lb. per sq. in. An average tensile stress of more than 300 lb. per sq. in. is developed in the columns having the stronger concrete before equilibrium is reached. If cracking occurred in these columns it is probably of little importance.

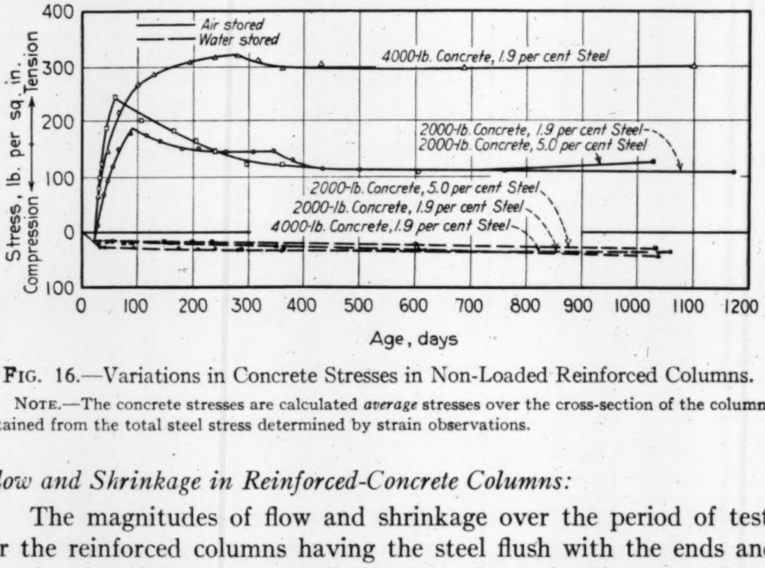

FIG. 16.—Variations in Concrete Stresses in Non-Loaded Reinforced Columns.

NOTE.—The concrete stresses are calculated *average* stresses over the cross-section of the columns. obtained from the total steel stress determined by strain observations.

Flow and Shrinkage in Reinforced-Concrete Columns:

The magnitudes of flow and shrinkage over the period of test, for the reinforced columns having the steel flush with the ends and also for the plain concrete cylinders, are shown in Figs. 17 and 18. The data are based upon the observed deformation in the concrete. It was found that for the columns with the steel flush with the ends the deformations of the steel and the concrete were practically the same.

Figure 17 shows the changes in length of the plain and the reinforced-concrete specimens, due to plastic flow, over the period of observation. In the case of concretes having comparable initial stresses due to application of load, the influence of reinforcement in reducing the flow is apparent. For example, for the dry-storage condition, the

2000-lb. concrete with no steel exhibits a flow about 30 per cent greater than the flow of the corresponding concrete reinforced with 1.9 per cent steel; and the flow of the 4000-lb. concrete having no steel is about 50 per cent greater than the flow of the corresponding concrete having 1.9 per cent of steel. Similar tendencies are exhibited by the water-stored concretes, although the magnitudes of flow are approximately one-third of the magnitudes of flow in the air-stored concretes.

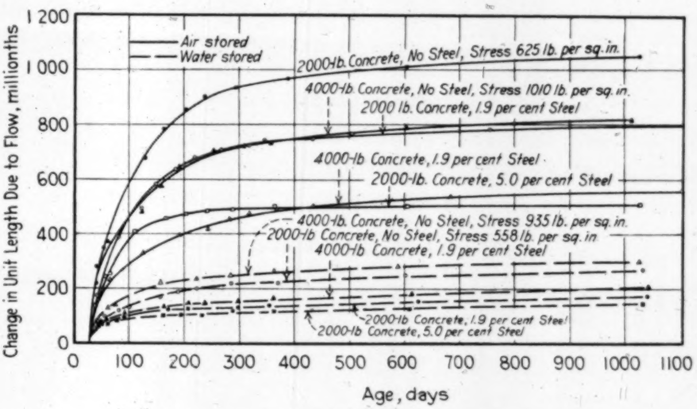

FIG. 17.—Changes in Length of Concrete Columns Due to Plastic Flow.

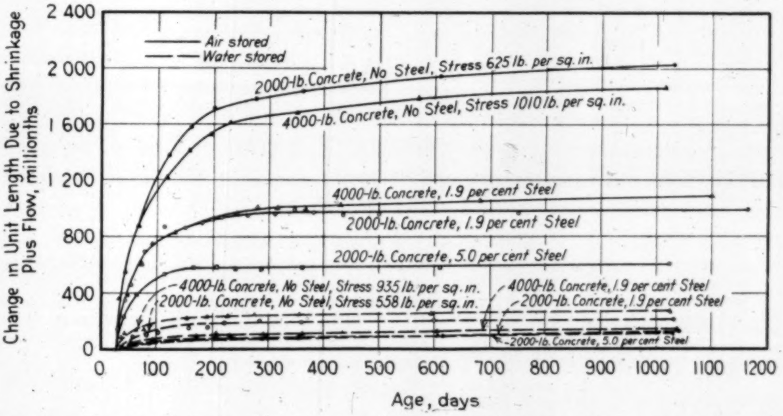

FIG. 18.—Changes in Length of Concrete Columns Due to Plastic Flow and Shrinkage Combined.

In Fig. 18 are given the total changes in length over the period of observation, due to flow and shrinkage combined. Here it will be noted that for given amounts of reinforcing steel the deformations for the air-stored concretes of either quality are nearly the same. This is due to the fact that although the concrete of richer mix exhibits less flow, it shrinks more. These diagrams are of particular interest in indicating the magnitude of total deformation due to time effects. After a period of 1000 days in air, the plain concrete columns have shortened axially an amount equivalent to about 2.4 in. per 100 ft., the concrete columns reinforced with 1.9 per cent of steel have shortened the equivalent of about 1.2 in. per 100 ft., and the columns reinforced with 5.0 per cent of steel have shortened the equivalent of about 0.7 in. per 100 ft. The columns stored under water exhibit deformations, over the same period, equivalent to less than 0.4 in. per 100 ft.

Slip Between Concrete and Steel in Columns in Which the Reinforcing Is Short of the Ends of the Columns:

As previously indicated, in the columns having the steel short of the ends the load was transferred to the steel entirely through bond with the concrete. The purpose of this group of tests was to determine the stress distribution in reinforced-concrete columns in which there may occur *slip* or *flow in bond.*

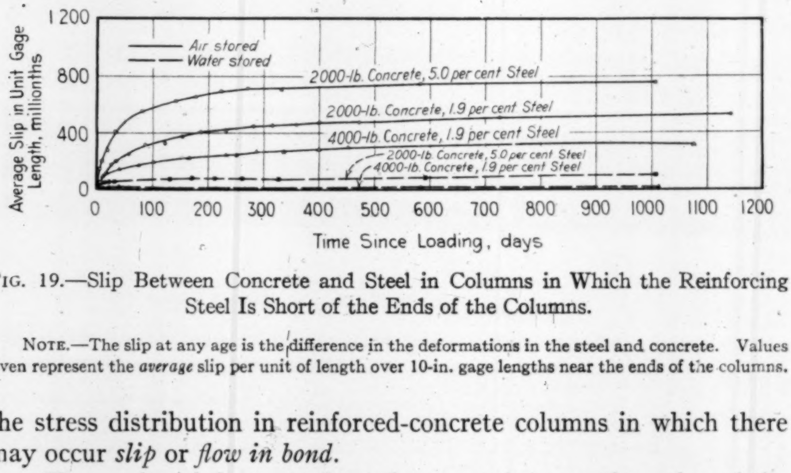

FIG. 19.—Slip Between Concrete and Steel in Columns in Which the Reinforcing Steel Is Short of the Ends of the Columns.

NOTE.—The slip at any age is the difference in the deformations in the steel and concrete. Values given represent the *average* slip per unit of length over 10-in. gage lengths near the ends of the columns.

The results of the tests show that over the central portion of the length of the columns the deformations are the same in the steel and in the concrete, and these deformations are of the same magnitude as in the columns having the steel flush with the ends. However, observations over 10-in. gage lengths near the ends of the loaded columns show appreciable differences in deformations of the steel and the concrete. In every case the concrete, over a given period of time, deforms more than the embedded steel, indicating a slow yielding in bond. This differential movement of the concrete with respect to the steel is herein termed *slip.*

The increase, with respect to time, of the average slip per unit gage length is shown for the various types of columns in Fig. 19. While the slip per inch undoubtedly increases rapidly near the ends of the reinforcing bars, the average slip observed over the 10-in. length gives a fair indication of the magnitude of the relative movements of the steel and concrete. For example, the slip after 1000 days in the columns made of 2000-lb. concrete and having 5.0 per cent steel is about 750 millionths in. per inch, which is of approximately the same magnitude as the total deformation of the steel itself.

As indicated in the figure, the air-stored columns made of 2000-lb. concrete and having 5.0 per cent steel exhibit after 1000 days under load a slip about 50 per cent greater than the slip of corresponding columns with 1.9 per cent steel. With the same percentage of steel, the columns made of the 4000-lb. concrete show slip of about two-thirds of that for columns made of 2000-lb. concrete. The slip in the water-stored columns is considerably less than that in the columns stored in air. In the water-stored columns made of 4000-lb. concrete and having 1.9 per cent steel, the slip is practically zero.

CONCLUSIONS

The following conclusions regarding the behavior of concrete under sustained compressive stress and regarding the effects of the deformations that take place with time are based upon a study of the results of observations made to date. The tests should be considered, however, as still in progress. Findings made in tests already carried out have pointed to the desirability of further tests, which are at present projected.

1. The major portion of the plastic flow or time-yield in concrete under sustained compressive stress is apparently due to seepage of colloidal water from the cement gel (Figs. 1, 2, 3, 4).

2. So far as gain or loss of moisture is concerned, plastic flow and shrinkage are intimately related (Figs. 1, 2, 3, 4).

3. The moisture content of the cement gel has a marked effect upon the seepage and hence upon the time-yield of concrete. The flow of a totally dry concrete is of very small magnitude (Fig. 3).

4. The rate or extent of hydration of the cement alters the time-flow relation appreciably, as indicated by the effect of age at time of loading (Fig. 9).

5. It is possible that in addition to seepage, both crystalline and viscous flow may take place to some extent. Crystalline flow is suggested by differences in flow observed in concrete containing different mineral aggregates (Fig. 6). Viscous flow, and perhaps crystalline

flow, is suggested by the existence of lateral flow accompanying axial flow when the lateral dimensions are unrestrained (Fig. 8).

6. When differences in flow may be due to causes other than seepage, the relative shrinkage of different concretes (concretes containing different mineral aggregates) is no criterion of the relative flows of those concretes (Fig. 6).

7. The gradation of the aggregate appreciably alters the time-flow relation for otherwise similar concretes (Fig. 5). The concrete having aggregate of coarser gradation exhibits greater flow.

8. The magnitude of flow is affected by the size of the mass. Within limits of the tests it is found that the larger the mass the less the flow (Fig. 7). Thus in general, the results of flow tests on small specimens are not directly applicable in estimating the probable time effects in large concrete masses, although flow of appreciable magnitude may possibly occur in large masses as is indicated by tests of concrete specimens maintained under load in sealed containers and restrained from lateral movement (Fig. 10).

9. If the moisture conditions are constant, temperature appears to have little or no effect upon flow. Observed differences in flow, for air-stored concretes at various temperatures, may be attributed to differences in the moisture content of the air at the given temperatures (Fig. 4).

10. The higher the sustained stress, the greater the flow. Under the given test conditions for concretes loaded at ages of 28 days and 3 months, and for stresses ranging from 300 to 900 lb. per sq. in., it is found that the flow is roughly proportional to the sustained stress, but outside the given limits this relation does not hold (Fig. 9, Table I).

11. In plain concretes, under certain conditions, flow is observed to continue even after almost 7 years under sustained stress, although the *rate* of flow is exceedingly small. In most cases the flow practically ceases after 1 or 2 years at the most (Figs. 1, 2, 6, 9, 17, 18).

12. In concretes stored in dry air the shrinkage acts with plastic flow to increase the total change in length with time. In many cases the magnitude of the shrinkage is practically equal to that of the flow for concretes at ordinary working stresses, say 800 lb. per sq. in. (Figs. 1, 2, 4, 6).

13. When plain concrete cylinders of relatively small diameters (4 in.) are stored in dry air under sustained stresses, within the range of ordinary working stresses for concrete, the total change in length due to flow and shrinkage combined may in time reach a magnitude equivalent to 1 or 2 in. per 100 ft. (Figs. 1, 2, 9).

14. For plain concrete cylinders stored under moist conditions,

the total deformation due to flow and expansion may be of the order of one-third to one-fourth of the total deformation of similar concretes stored in dry air (Figs. 1, 2, 4).

15. The flow and shrinkage may vary appreciably throughout a mass of concrete, especially when it is exposed to drying conditions (Fig. 8). This fact points to the existence of considerable differences between the assumed and the actual distribution of stress across a section in concrete members of structural dimensions.

16. After release of sustained load, a plastic recovery takes place which may continue over a period of several weeks and which may reach an appreciable fraction of the flow produced by the previously sustained load (Figs. 11, 12, 13). The phenomenon is probably due, in part, to the resilience of the compressed aggregate particles. Recovery is observed to take place even after loads have been sustained for almost 7 years (Fig. 11).

17. Although steel reinforcement in concrete columns is effective in reducing the flow and shrinkage below that which takes place in plain concrete columns, nevertheless deformations of appreciable magnitude may take place over a period of time. In the air-stored reinforced columns for which tests are reported here, the total unit time-deformations are of the order of one-half to one-third of those in corresponding air-stored plain concrete columns (Figs. 17, 18).

18. Appreciable changes in the distribution of stress between the steel and concrete take place in reinforced columns under load in dry air. In the tests here reported, in which the columns were subjected to the maximum load allowed by specifications, the stress in the steel due to all causes reaches, for certain conditions, a magnitude of 40,000 lb. per sq. in. (Fig. 14 (*a*)). This fact points to the desirability of the use of at least a medium grade of reinforcing steel for concrete compression members, particularly when the member is to be subjected to drying conditions. The use of coatings which prevent the loss of moisture seems to merit consideration as a means of reducing the development of high steel stresses in reinforced concrete compression members. The increase in steel stress with time, in reinforced concrete columns under load and stored under water, is considerably less than the increase in stress of corresponding columns stored in dry air. Under the conditions of test, the maximum stress due to all causes reaches a value of 12,000 lb. per sq. in. (Fig. 14 (*b*)).

19. The reduction in concrete stress with time, corresponding to the increase in steel stress, may be very marked. It is found that in some columns under test in dry air, in spite of the continued application of the full load which is permitted under the design specifications,

the concrete stress is gradually reduced to practically zero. For columns stored under water, the reduction in concrete stress is much less marked (Fig. 15).

20. In reinforced concrete columns not under load and stored in air, concrete stresses due to shrinkage may be such that the concrete will fail in tension, after which the development of cracks relieves the stresses set up in both the steel and concrete (Fig. 16).

21. In compression members in which the load is transferred to the steel solely through bond with the concrete, a considerable slip or flow in bond between steel and concrete may take place over a period of time. This condition is most marked when a relatively lean, or weak, concrete is reinforced with a high percentage of steel (Fig. 19). The tests indicate that slip occurs over only a relatively short distance near the ends of the steel reinforcing bars.

22. The rate of time-yield in reinforced-concrete columns stored in dry air appears to become practically zero after a period of about one year. In water-stored columns, equilibrium is evidently reached within a few months.

TESTS ON REINFORCED BRICK MASONRY COLUMNS

By M. O. Withey[1]

Synopsis

Tests of thirty-two 12½ by 12½-in. brick columns 6 ft. high made with Chicago common and with Waupaca face brick are reported. The variables included three percentages of longitudinal and a like variation in lateral reinforcement together with two high-strength portland cement lime mortars. The results indicate that the column strength varies directly with the strength of the masonry and the percentage of longitudinal steel, and is increased by the use of lateral reinforcement. Strengths of 5000 lb. per sq. in. based on gross area and over 7000 lb. per sq. in. on the cores after shell spalling were attained. For suitable reinforced brick masonry columns a factor of safety of 3.5 to 4.0 is suggested.

Introduction

This series of tests is the outgrowth of a program planned by the Reinforced Brick Masonry Board to determine the properties of reinforced brick masonry columns. The tests were conducted in the Materials Testing Laboratory at The University of Wisconsin under the direction of the author during the summer of 1933. Effective assistance was rendered by K. F. Wendt and L. O. Hanson, Instructors in Mechanics, and L. M. Basford, Technician. Acknowledgment is made to Con Gmeiner and Sons, Inc., of Waupaca, and the Illinois Brick Co. of Chicago for donations of brick. Funds for conducting the tests were donated to the university from the Research Fund of the Reinforced Brick Masonry Research Board.

Materials

Cement.—Briquets of 1:3 standard sand mortar made from the cement used in these tests exhibited a tensile strength of 333 and 388 lb. per sq. in. at 7 and 28 days, respectively. The compressive strength of 2 by 4-in. cylinders of the same mix was 2880 and 3970 lb. per sq. in. at the same ages.

Sand.—The Janesville mason's sand weighed 106 lb. per cu. ft. All of it passed a No. 8 sieve, 71 per cent passed a No. 28 sieve and 17 per cent passed a No. 48 sieve. The average fineness modulus was 2.17.

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Steel.—The intermediate grade 1-in. round deformed bars used for longitudinal reinforcement had tensile and compressive yield points of 47,840 anf 48,720 lb. per sq. in., respectively. The yield point of the $\frac{3}{8}$-in. round steel rods used in the hoops was 53,800 lb. per sq. in. The strength of the fillet and spot welds in the 10-in. hoops after heating to 900 C. and straightening while hot averaged 58,900 lb. per sq. in. of rod cross-section. Spot welds alone on the hoops were unsatisfactory.

Brick.—The average percentages of absorption for the Chicago common clay brick were 3.6 at 1 min., 7.0 at 30 min., 10.7 at 48 hr., and 13.9 after a 5-hr. boil. These brick were 8.15 by 3.64 by 2.21 in., weighed 4.23 lb., had a modulus of rupture of 930 lb. per sq. in. and a crushing strength flatwise of 2735 lb. per sq. in. The average percentages of absorption for the Waupaca clay face brick were 0.7 at 1 min., 1.5 at 30 min., 2.2 at 48 hr., and 3.2 after a 5-hr. boil. Their dimensions were 7.52 by 3.42 by 2.18 in. They weighed 4.26 lb. and had a modulus of rupture of 2120 lb. per sq. in. Bedded flatwise in plaster of Paris, half brick had a crushing strength of 14,420 lb. per sq. in., bedded in Lumnite the flatwise crushing strength was 20,000 lb. per sq. in.

Mortar.—All mortar proportions are expressed in terms of dry weight of dolomitic hydrated lime, L, cement, C, and sand, S. Amounts of lime, cement, and sand sufficient for two hours work were weighed, placed in a mortar box, and dry mixed until of uniform appearance. Sufficient water was then added to make a plastic mortar with a flow of 80 to 100 as determined on a 10-in. flow table using thirty 0.18-in. drops in 30 sec. The water-cement ratio, by weight, for the $1L{:}3C{:}12S$ averaged 0.88 and for the $1L{:}3C{:}7.5S$, 0.69. Occasional retempering was permitted when the batch in the mortar box became too stiff. The mortar was mixed by an experienced mason's helper. Specific weights for moist-cured mortars of $1L{:}3C{:}12S$ and $1L{:}3C{:}7.5S$ were 136 and 135 lb. per cu. ft., respectively.

CONSTRUCTION

Columns.—All columns and auxiliary brick prisms were built by an experienced mason on leveled cast-iron base plates. Like columns were in most cases made on different days. Just prior to laying, Chicago brick were dumped from a wheel barrow onto the floor and wetted with a hose. From determinations, each on five bricks on three different days, it was found that the water content of the bricks when laid was between 5 and 7 per cent, by weight. Waupaca brick were laid air-dry and probably did not contain over 0.2 per cent of water. Joints were struck flush with the surface of the work. Figure 1 shows column details. Owing to the small space inside of the vertical rods, only small bats could be used in filling this space and the proportion of mortar was much higher than in other parts of the columns. Due to the small size of the Waupaca brick and the use of a fixed spacing for the vertical rods the vertical joints in these columns were often $1\frac{1}{4}$ in. wide, a condition which undoubtedly weakened the columns.

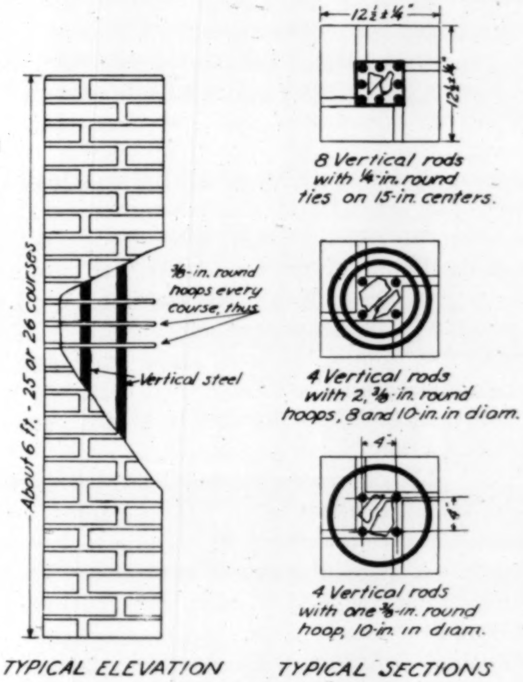
12½ ± ¼"
12½ ± ¼"
8 Vertical rods with ¼-in. round ties on 15-in. centers.
⅜-in. round hoops every course, thus
Vertical steel
About 6 ft. - 25 or 26 courses
4 Vertical rods with 2, ⅜-in. round hoops, 8 and 10-in. in diam.
4"
4"
4 Vertical rods with one ⅜-in. round hoop, 10-in. in diam.
TYPICAL ELEVATION
TYPICAL SECTIONS

FIG. 1.—Column Details.

Columns were cured under damp canvas wetted twice a day for one week and thereafter in the air of the laboratory until tested.

Mortar Specimens.—From every batch of mortar used in making columns, three 2-in. cubes and three 2 by 4-in. cylinders were made. These were moist cured for 7 days and then stored under dry canvas until the corresponding columns were tested, when they were also broken.

Brick Prisms.—One or two 8 by 8 by 16-in. brick prisms were made on each day that columns were built. These prisms were 6 courses high with the brick laid flatwise with ½-in. joints and headers and stretchers alternated in every other course. They were cured in the same manner as the columns and tested at the same age.

TESTING

Columns.—The columns were tested when approximately 4 weeks old in a 600,000-lb. hydraulic testing machine provided with a spherical upper head, see Table VI. Three of the strongest columns were broken in the two-screw 1,000,000-lb. testing machine at the Forest Products Laboratory. Longitudinal strains were measured over a 50-in gage length by wire-wound dial compressometers reading to 0.0001 in.

Loads were applied in increments of 100 to 330 lb. per sq. in., depending upon the strength of the column under test. Strain readings were taken 30 sec. to 1 min. after application of the load, depending upon the time required for cessation of motion of the dial pointers. The rate of stressing near the maximum load was usually between 150 and 250 lb. per sq. in. per min.

In a considerable number of tests, loads approximating a third to a half of the ultimate for the column were repeated several times or held for a half hour or longer in order to secure some data on the plastic flow of the column.

Brick Prisms.—The prisms were tested in a three-screw 250,000-lb. machine using a spherical upper head. Deformations were obtained with a wire-wound dial compressometer reading to 0.0001 in. over a 10-in. gage length. A speed of movable head of 0.06 in. per min. for the machine running idle was used. Loads were applied in increments of 10,000 to 25,000 lb.

Mortar Prisms.—Cylinders and cubes were capped with plaster of Paris and tested at a speed of 0.03 in. per min.

AUXILIARY TESTS

Mortar Prisms.—The average results of the tests on the mortar and the brick prisms are given in Table I. The ratio, strength of cylinder to strength of cube, for the $1L:3C:12S$ mix averaged 0.99 and for the $1L:3C:7.5S$ mix, 1.03. Hence, both cube and cylinder strengths were averaged and reported in column 6 of Table I. With one exception, the strengths of the batches of $1L:3C:12S$ mortar were all within 18 per cent of the average, 2335 lb. per sq. in. For the $1L:3C:7.5S$ batches the strengths were within 6 per cent of the average, 3567 lb. per sq. in.

Brick Prisms.—Prisms made with Chicago brick and $1L:3C:12S$ mortar all had strengths within 8 per cent of the average, 1040 lb. per sq. in. Failure usually began by transverse rupture of a brick at a vertical mortar joint. This was followed by extension of the crack vertically, splitting of the prisms into smaller units, and crushing of one or more bricks. No failures were due to weakness of mortar. Values for the secant modulus of elasticity at one-third the ultimate strength were, however, far more variable, ranging from 610,000 lb. per sq. in. to 1,120,000 lb. per sq. in. and averaging 905,000 lb. per sq. in. Why these prisms exhibited lower stiffnesses than the corresponding plain columns is not definitely known. On the other hand, these prisms exhibited a stiffness in direct proportion to their strength Waupaca brick prisms made with $1L:3C:12S$ mortar varied little from the average, 3675 lb. per sq. in. There was evidence in two of the three tests of spalling at the mortar joints after transverse rupture of a brick.

TABLE I.—AVERAGE COMPRESSIVE STRENGTHS AND MODULI OF ELASTICITY FOR AUXILIARY 8 BY 8 BY 16-IN. PRISMS.

Age at Test, 4 weeks.

Column	Mortar Mix by Weight, lime: cement: sand	Kind of Brick	Modulus of Elasticity, lb. per sq. in.	Compressive Strength, lb. per sq. in.			Ratios		
				Mortar (M)	Brick (B)	Prisms (P)	P/M	P/B	M/B
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
Nos. 1 to 16....	1:3:12	Chicago	905 000	2310	2 734	1040	0.45	0.38	0.85
Nos. 17 to 20....	1:3:12	Waupaca	2 680 000	2375	14 420	3675	1.55	0.25	0.16
Nos. 21 to 32....	1:3:7.5	Waupaca	3 680 000	3567	14 420	4521	1.27	0.31	0.25

Waupaca brick prisms made with $1L:3C:7.5S$ mortar varied less than 12 per cent from the average 4521 lb. per sq. in. Evidence of spalling of mortar prior to failure was noted in one instance at a load of 4200 lb. per sq. in. In general, however, these prisms failed in the same manner as the Chicago brick prisms but with far less warning and much greater violence. From the P/B and M/B ratios in Table I, it appears likely that still stronger prisms and columns can be made with Waupaca brick by using a mortar of higher compressive strength than the $1L:3C:7.5S$.

RESULTS OF TESTS

Behavior of Columns Under Test.—The properties of the 32 columns tested are listed in Table II. Typical stress-strain curves are shown in Fig. 2. The tests of the plain columns were characterized by

TABLE II.—PROPERTIES OF REINFORCED BRICK COLUMNS.[a]

Horizontal mortar joints were $\frac{1}{2}$-in. thick. Hoops had 2-in. lap joints. Joints in all 8-in. hoops and in 10-in. hoops of columns Nos. 7, 9, 11, 13, 15 were spot welded. All other 10-in. hoops were lap welded.

Column	Reinforcement	Gross Area, sq. in.	Core Area, sq. in.	Percentage of Reinforcement				Kind of Brick	Mortar Mix by Weight, lime: cement: sand
				Longitudinal, Based on Area		Lateral, Based on Area			
				Gross	Core	Gross	Core		
No. 1	None	157.5	....	0	....	0	....	Chicago	1:3:13.5
No. 2		160.6	....	0	....	0	....	Chicago	1:3:12
No. 3	Four 1-in. round deformed rods with $\frac{1}{4}$-in. ties on 15-in. centers	162.0	....	1.92	....	0	....	Chicago	1:3:13.5
No. 4		161.3	....	1.93	....	0	....	Chicago	1:3:12
No. 5	Eight 1-in. round deformed rods with $\frac{1}{4}$-in. ties on 15-in. centers	163.2	....	3.81	....	0	....	Chicago	1:3:13.5
No. 6		162.6	....	3.83	....	0	....	Chicago	1:3:12
No. 7	Two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	162.6	90.8	0	0	1.36	2.44	Chicago	1:3:12
No. 8		156.8	90.8	0	0	1.37	2.37	Chicago	1:3:12
No. 9	Four 1-in. round deformed rods with two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	165.8	90.8	1.88	3.42	1.32	2.41	Chicago	1:3:12
No. 10		163.2	90.8	1.90	3.42	1.34	2.41	Chicago	1:3:12
No. 11	Eight 1-in. round deformed rods with two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	161.3	90.8	3.86	6.85	1.36	2.42	Chicago	1:3:12
No. 12		163.8	90.8	3.80	6.85	1.33	2.41	Chicago	1:3:12
No. 13	Four 1-in. round deformed rods with one $\frac{3}{8}$-in. round hoop 10 in. in diameter at 2$\frac{1}{2}$-in. centers	160.0	90.8	1.94	3.42	0.75	1.33	Chicago	1:3:12
No. 14		161.3	90.8	1.93	3.42	0.75	1.33	Chicago	1:3:12
No. 15	Eight 1-in. round deformed rods with one $\frac{3}{8}$-in. round hoop 10 in. in diameter at 2$\frac{1}{2}$-in. centers	162.5	90.8	3.83	6.85	0.74	1.33	Chicago	1:3:12
No. 16		162.0	90.8	3.84	6.85	0.75	1.33	Chicago	1:3:12
No. 17	None	144.7	....	0	....	0	....	Waupaca	1:3:12
No. 18		144.5	....	0	....	0	....	Waupaca	1:3:12
No. 19	Eight 1-in. round deformed rods with $\frac{1}{4}$-in. ties on 15-in. centers	150.0	....	4.15	....	0	....	Waupaca	1:3:12
No. 20		151.9	....	4.10	....	0	....	Waupaca	1:3:12
No. 21	Two $\frac{3}{8}$-in. round hoops 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	148.8	90.8	0	0	1.54	2.53	Waupaca	1:3:7.5
No. 22		150.7	90.8	0	0	1.51	2.51	Waupaca	1:3:7.5
No. 23	Four 1-in. round deformed rods with two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	150.7	90.8	2.07	3.42	1.51	2.51	Waupaca	1:3:7.5
No. 24		150.7	90.8	2.07	3.42	1.52	2.52	Waupaca	1:3:7.5
No. 25	Eight 1-in. round deformed rods with two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$ in. centers	152.0	90.8	4.10	6.85	1.50	2.51	Waupaca	1:3:7.5
No. 26		154.5	90.8	4.03	6.85	1.47	2.51	Waupaca	1:3:7.5
No. 27	Four 1-in. round deformed rods with two $\frac{3}{8}$-in. round hoops, 8 and 10 in. in diameter at 2$\frac{1}{2}$-in. centers	156.8	90.8	1.98	3.42	1.48	2.56	Waupaca	1:3:7.5
No. 28		159.4	90.8	1.95	3.42	1.46	2.56	Waupaca	1:3:7.5
No. 29		152.5	90.8	2.04	3.42	1.52	2.56	Waupaca	1:3:7.5
No. 30	None	162.0	....	0	....	0	....	Waupaca	1:3:7.5
No. 31	Same as columns Nos. 9 and 10[b]	158.4	90.8	1.96	3.42	1.41	2.46	Waupaca	1:3:7.5
No. 32	Same as columns Nos. 13 and 14[b]	154.6	90.8	2.01	3.42	0.80	1.36	Waupaca	1:3:7.5

[a] All columns were approximately 6 ft. high excepting columns Nos. 27, 28, and 29 which were 12 ft. high.

[b] Made with concrete cores. Compressive strength of concrete, 5340 lb. per sq. in.

barely audible creaking sounds at loads approximating half the ultimate. At loads near the ultimate, transverse cracks appeared in the brick usually at vertical joints. These cracks spread vertically through bonding planes of vertical joints and brick and an abrupt failure ensued. Figure 3 shows the failure of column No. 17.

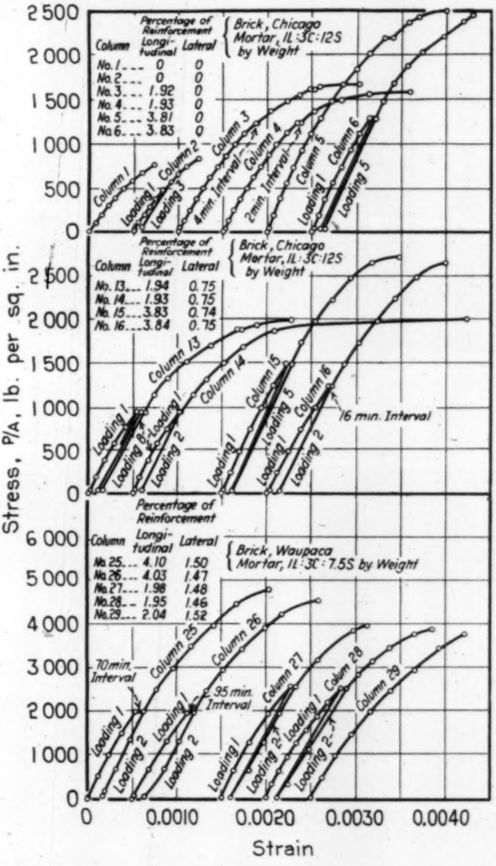

FIG. 2.—Stress-Strain Curves for Columns Nos. 1 to 6, 13 to 16, and 25 to 29.

Columns equipped with ¼-in. ties centered 15 in. apart after being strained beyond the yield point of the vertical steel also failed suddenly with separations of the outer shell from the core as shown for column No. 19 in Fig. 4.

Lateral hoops in every joint served to increase markedly the toughness and to some extent the strengths of the columns. Failures

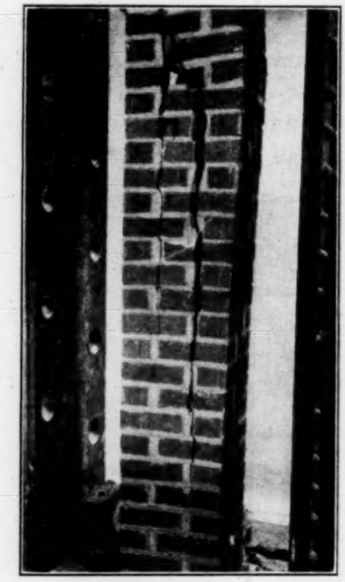

FIG. 3.—Failure of Plain Waupaca Brick Column No. 17.

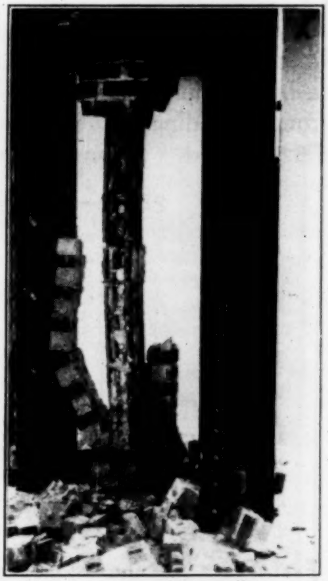

FIG. 4.—Failure of Waupaca Brick Column No. 19. Reinforced with 4 per cent of vertical steel.

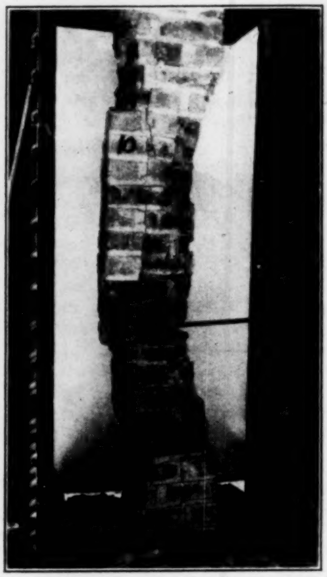

FIG. 5.—Failure of Chicago Brick Column No. 10. Reinforced with hoops and vertical steel.

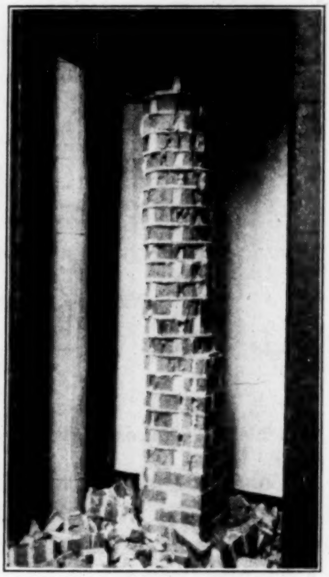

FIG. 6.—Failure of Waupaca Brick Column No. 25, the Strongest Tested.

of hooped columns were accompanied by spalling of the portions of brick outside of the outer ring and considerable longitudinal strain and deflection before failure. In tests of hooped columns Nos. 10, 15, 16, 21, 23, 25, and 26, shell spalling was accompanied by a decided drop in load which was followed by a definite pick-up until the core strength was reached. In several tests, these maximum core loads were held for 1 to 10 min. before a weld parted or the masonry failed. In none of the tests did the maximum core load (P_1) exceed the previous maximum (P) which the column had held prior to spalling of the shell. Typical failures of this type of column are shown in Figs. 5 and 6.

TABLE III.—COMPARISON OF STRENGTHS OF COLUMNS AND AUXILIARY PRISMS.

Column	Percentage of Reinforcement		Maximum Load, lb.	Load at Failure of Core, lb.	Ultimate Strength, lb. per sq. in.			Ratios		
					Column		Prism			
	Longitudinal	Lateral	(P)	(P_1)	Based on Gross Area (p)	Based on Core Area (p_1)	(q)	p/q	p_1/q	p_1/p
No. 1	0	0	154 800	......	983	....	1040	0.94	....	....
No. 2	0	0	174 900	......	1090	....		1.05	....	....
No. 3	1.92	0	270 000	......	1667	....		1.60	....	....
No. 4	1.93	0	255 000	......	1580	....		1.52	....	....
No. 5	3.81	0	412 300	......	2525	....		2.43	....	....
No. 6	3.83	0	428 800	......	2640	....		2.54	....	....
No. 7	0	1.36	218 000	190 000	1340	2090		1.29	2.01	1.56
No. 8	0	1.37	175 400	143 000	1120	1575		1.08	1.51	1.41
No. 9	1.88	1.32	341 000	323 000	2060	3560		1.98	3.42	1.73
No. 10	1.90	1.34	333 500	320 500	2040	3530		1.96	3.39	1.73
No. 11	3.86	1.36	467 000	378 000	2900	4160		2.79	4.00	1.44
No. 12	3.80	1.33	453 000	......	2765	....		2.66	....	....
No. 13	1.94	0.75	343 500	......	2145	....		2.06	....	....
No. 14	1.93	0.75	322 200	269 000	2000	2970		1.92	2.85	1.485
No. 15	3.83	0.74	461 750	420 000	2840	4630		2.73	4.45	1.63
No. 16	3.84	0.75	460 400	389 250	2840	4300		2.73	4.14	1.51
No. 17	0	0	361 500	......	2500	....	3675	0.68	....	....
No. 18	0	0	377 500	......	2615	....		0.71	....	....
No. 19	4.15	0	612 700	......	4080	....		1.11	....	....
No. 20	4.10	0	631 500	......	4160	....		1.13	....	....
No. 21	0	1.54	581 000	456 000	3900	5030	4521	0.86	1.11	1.29
No. 22	0	1.51	564 600	449 000	3740	4950		0.83	1.09	1.32
No. 23	2.07	1.51	608 000	560 500	4040	6190		0.89	1.37	1.53
No. 24	2.07	1.52	605 500	538 500	4020	5930		0.89	1.31	1.47
No. 25	4.10	1.50	768 700	694 000	5050	7650		1.12	1.69	1.52
No. 26	4.03	1.47	745 000	585 000	4820	6450		1.07	1.43	1.34
No. 27	1.98	1.48	620 000	515 500	3950	5690		0.87	1.26	1.44
No. 28	1.95	1.46	645 000	484 000	4045	5340		0.89	1.18	1.32
No. 29	2.04	1.52	591 500	510 500	3880	5620		0.86	1.24	1.45
No. 30	0	0	542 000	......	3340	....		0.74	....	....
No. 31	1.96	1.41	620 000	565 500	3915	6225		0.87	1.38	1.59
No. 32	2.01	0.80	712 200	440 500	4610	4850		1.04	1.07	1.05

Influence of Various Factors on Column Strength.—A comparison of the strengths of columns and auxiliary prisms is given in Table III. Examination of these data shows that the maximum deviation from the average strength for a given pair of columns was 9 per cent. In all but two pairs, the deviation from the average was less than 3 per cent. For columns Nos. 23, 24, 27, 28 and 29, which differed only in length, the greatest deviation from the average strength was only 2.7 per cent and the average deviation was under 1.5 per cent.

The average strength of the auxiliary prisms of Chicago brick checked very closely the strengths of the two plain columns made of that brick. With the Waupaca brick, however, the p/q ratios show that the plain columns made with that brick carried only 68 to 74 per cent of the maximum stress supported by the corresponding prisms. Part of these deficiencies of the Waupaca brick columns can be ascribed to weaknesses induced by the wide vertical joints and the inferior lateral bond which existed in the columns due to method of laying. The use of hoops in each joint of columns Nos. 21 and 22 partly overcame this deficiency and increased the p/q ratio to 0.84 for these columns. The void spaces around the bats were another source of weakness in these columns. Despite these defects, with the same mortar the plain columns with Waupaca brick were $2\frac{1}{2}$ times as strong as those made with Chicago brick and with $1L:3C:7.5S$ mortar were $3\frac{1}{3}$ times as strong.

Considering plain columns made with $1L:3C:12S$ mortar the strengths were approximately in proportion to the moduli of rupture of the brick. Comparison of the ratios p_1/q, p/q, and p_1/p in Table III shows that the core material when fully stressed was decidedly stronger than average strength of the column or prism. Considerations of these data together with the inherent weaknesses of these cores due to fabrication difficulties indicate that outer hoop diameters should be made as large as limits of proper protection for the hoops against weathering or fire permit in order to secure high efficiency from the hoops.

Waupaca plain brick column No. 30 made with $1L:3C:7.5S$ mortar was over 30 per cent stronger than columns Nos. 17 and 18 made with $1L:3C:12S$ mortar.

Columns made with either brick were strengthened in direct proportion to the amount of included vertical steel.

Columns of Chicago brick reinforced with two $\frac{3}{8}$-in hoops per joint exhibited about 25 per cent greater strength than similar plain columns. The data for the Chicago brick columns indicate that a single $\frac{3}{8}$ by 10-in. hoop per joint in a $12\frac{1}{2}$ by $12\frac{1}{2}$ in. column is sufficient lateral reinforcement for the ordinary proportions of vertical steel.

In the Waupaca brick columns the use of two $\frac{3}{8}$-in. hoops per joint effected an increase of 14 per cent over plain column strength. A fair comparison between Waupaca columns Nos. 32 and 31, reinforced with one and two hoops per joint, respectively, cannot be made owing to defective filling of the concrete core at the lower end of column No. 31. Furthermore, two of the bars in column No. 32 had yield points 10,000 lb. per sq. in. higher than the bars used in the other columns tested.

Column cores made of a given brick after spalling of shells exhibited strengths in direct proportion to the percentage of vertical steel. The effect of a given percentage of steel (based on the core area) on core strength (p_1) was about the same as the effect of the same percentage (based on gross area) on the ultimate strength (p).

TABLE IV.—UNIT STRESSES CARRIED BY BRICKWORK AND BY STEEL AT MAXIMUM LOAD.

Column	Percentage of Reinforcement		Unit Stresses, lb. per sq. in.					Ultimate Strength of Prisms, lb. per sq. in.	Ratios	
			Based on Gross Area	Based on Core Area	Steel	Brickwork				
	Longitudinal	Lateral	(p)	(p_1)	(y)	Based on Gross Area (b)	Based on Core Area (b_1)	(q)	b/q	b_1/q
No. 1	0	0	983	....	......	983	....	1040	0.95	....
No. 2	0	0	1090	....	......	1090	....		1.05	....
No. 3	1.92	0	1667	....	48 720	743	....		0.71	....
No. 4	1.93	0	1580	....		651	....		0.63	....
No. 5	3.81	0	2525	....		690	....		0.66	....
No. 6	3.83	0	2640	....		798	....		0.77	....
No. 7	0	1.36	1340	2090	......	1340	2090		1.29	2.01
No. 8	0	1.37	1120	1575	......	1120	1575		1.08	1.51
No. 9	1.88	1.32	2060	3560	48 720	1162	1950		1.12	1.87
No. 10	1.90	1.34	2040	3530		1133	1920		1.09	1.85
No. 11	3.86	1.36	2900	4160		1050	874		1.01	0.84
No. 12	3.80	1.33	2765	....		945	....		0.91	....
No. 13	1.94	0.75	2145	....		1221	....		1.18	....
No. 14	1.93	0.75	2000	2970		1076	1335		1.03	1.28
No. 15	3.83	0.74	2840	4630		1008	1370		0.96	1.32
No. 16	3.84	0.75	2840	4300		1002	1010		0.96	0.96
No. 17	0	0	2500	....	......	2500	....	3675	0.68	....
No. 18	0	0	2615	....	......	2615	....		0.71	....
No. 19	4.15	0	4080	....	48 720	2145	....		0.56	....
No. 20	4.10	0	4160	....		2250	....		0.61	....
No. 21	0	1.54	3900	5030	......	3900	5030	4521	0.86	1.11
No. 22	0	1.51	3740	4950	......	3740	4950		0.83	1.09
No. 23	2.07	1.51	4040	6190	48 720	3095	4655		0.68	1.03
No. 24	2.07	1.52	4020	5930		3070	4410		0.68	0.98
No. 25	4.10	1.50	5050	7650		3185	4605		0.70	1.02
No. 26	4.03	1.47	4820	6450		2975	3320		0.66	0.73
No. 27	1.98	1.48	3950	5690		3045	4150		0.67	0.92
No. 28	1.95	1.46	4045	5340		3155	3785		0.70	0.84
No. 29	2.04	1.52	3880	5620		2940	4090		0.65	0.90
No. 30	0	0	3340	....		3340	....		0.74	....
No. 31	1.96	1.41	3915	6225		3012	4715		0.67	1.04
No. 32	2.01	0.80	4610	4850		3700	3290		0.82	0.73

Owing to imperfect filling of the cores by the bats and mortar in the columns reinforced with vertical rods it is believed that the cores in these columns did not carry much of the load until the shell spalled. To test this assumption, columns Nos. 31 and 32 were built with cores poured of 5000 lb. per sq. in. concrete. Unfortunately, the mix was a bit too stiff and the lower end of column No. 31 was not perfectly filled and its strength was no greater than that of column No. 23 or No. 24. Column No. 32 gave much better results. Retests were then made on undamaged 3½-ft. lengths of columns Nos. 31 and 32. In the retest, column No. 31A had a strength of 4520 lb. per sq. in. at maximum load and carried for 10 minutes 6980 lb. per sq. in. on its core after spalling of the shell. Column No. 32A carried a load of 4880 lb. per sq. in. at maximum, but its core load was not well defined due to breaking of welds in hoops.

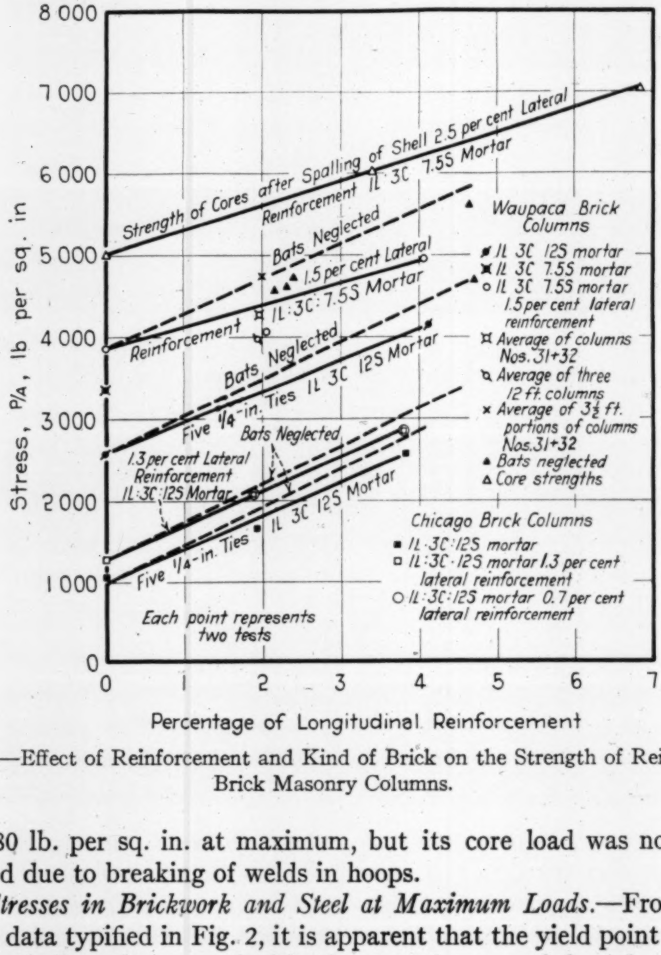

FIG. 7.—Effect of Reinforcement and Kind of Brick on the Strength of Reinforced Brick Masonry Columns.

Stresses in Brickwork and Steel at Maximum Loads.—From the strain data typified in Fig. 2, it is apparent that the yield point of the longitudinal steel was reached in the test of every reinforced column. Scaling of the longitudinal rods was also apparent in a number of tests. Since the stress-strain diagram for steel of the grade used is practically parallel to the strain axis for a considerable range in strain beyond the yield point, the stress in the vertical steel at the failure of these columns could not have been much in excess of the yield point. Assuming the steel stress at the column ultimate to be equal to the yield point, the stress in the brickwork was readily calculated and is entered in Table IV. A study of the b/q ratios in this table supports the belief that the cores were ineffective in carrying full stress at maximum load. The b_1/q ratios indicate that the restraining action of the hoops made it possible for the masonry within the hoops to carry a higher proportion of the prism strength than the column as a whole carried. There is one exception noted in b_1/q ratio (0.84) for column No. 11. This is due, in that column, to the breaking of a weld which occurred after the maximum load had been reached.

Relation of Strength to Percentage of Longitudinal Reinforcement.—The data for these tests showing the relation of unit loads to percentage of longitudinal steel have been plotted in Fig. 7. The percentages for the top curve in the figure are based on core areas, but all other percentages are based on gross areas of cross-sections. Full lines have been used to indicate the trends of the unmodified data. Dash lines show the strengths based on areas minus bats or strengths attainable with suitable concrete cores.

The equations of the dash lines marked "Bats Neglected" are of the form:

$$\frac{P}{A} = f_b\,(1\text{-}p) + p\,f_s + K\,p'\,f'_s \qquad \text{(1)}$$

where P = maximum load,
A = gross area of cross-section,
f_b = unit load for plain brick column,
p = longitudinal steel ratio in terms of gross area,
p' = lateral steel ratio in terms of gross area,
f_s = yield point of longitudinal steel,
f'_s = yield point of hoops, or weld strength if less than yield point, and
K = constant tentatively assumed to depend upon the ratio A/A_c and possibly on the kind of brick, where A_c = area of core within outer hoop.

For these tests with A/A_c approximating 1.7, f'_s is 54,000 lb. per sq. in, p' is 0.0075 for Chicago brick and 0.015 for Waupaca brick, and K is approximately 0.6. The validity of the last term of Eq. 1 is questionable until more data on the action of hoops has been secured.

Working Stresses.—The strain-repetition and set-repetition curves for these columns, together with the strain-time curves of others, see Fig. 8, indicate that the time-rate of strain is very small for loads of 35 to 55 per cent of the ultimate for the reinforced brick columns. In Table V are listed unit stresses in steel and brickwork at loads of one-fourth the maximum for the tested columns. ¶These were calculated by computing the steel stresses from strains by Hooke's law and subtracting these stresses from the total load to find the stresses in

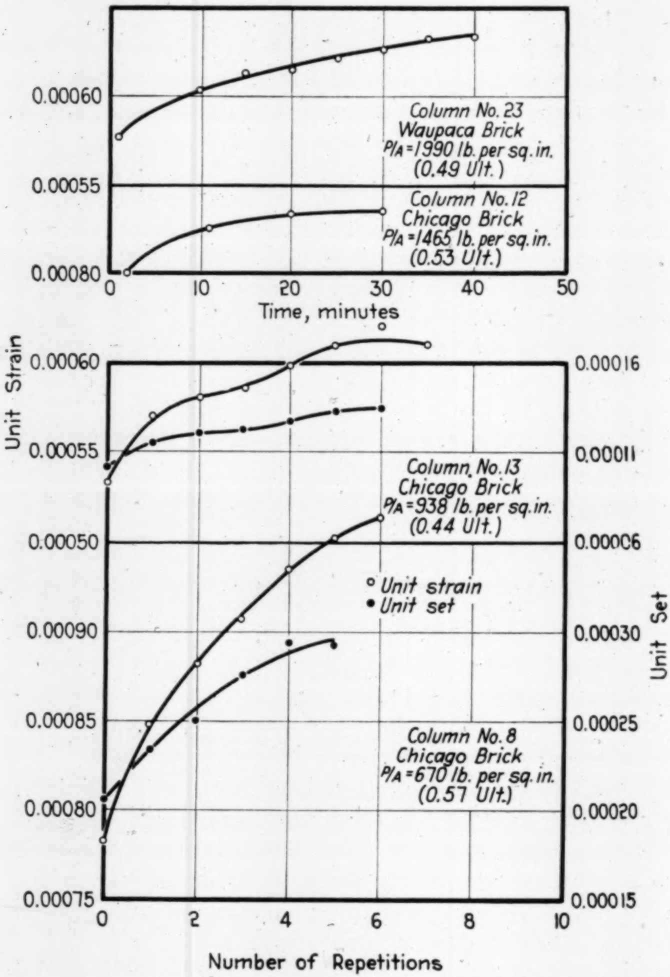

FIG. 8.—Strain-Time Curves for Columns Nos. 12 and 23 and Strain-Repetition and Set-Repetition Curves for Columns Nos. 8 and 13.

the brickwork. The ratios q/b' and y/s indicate the relation of these stresses to prism strengths and yield point of the longitudinal steel, respectively. The stresses in the brickwork for the reinforced columns assuming a factor of safety of 4 appear safe, especially with the accompanying high ratios of y/s. The latter would appear to be ample to avoid overstrain in the steel due to plastic flow. For the columns of the stronger brick reinforced with hoops and vertical rods, the factor of safety could be reduced to 3.5.

TABLE V.—UNIT STRESSES CARRIED BY BRICKWORK AND BY STEEL AT ONE-FOURTH MAXIMUM LOAD.

Column	Percentage of Reinforcement		Unit Stresses, lb. per sq. in.					Ratios	
	Longitudinal	Lateral	In Gross Area (p')	In Brickwork (b')	In Steel (s)	Prism Strength (q)	Yield Point of Steel (y)	q/b'	y/s
No. 1	0	0	246	246	.....	1040	48 720	4.23	....
No. 2	0	0	273	273	.....			3.81	....
No. 3	1.92	0	417	294	6 570			3.54	7.42
No. 4	1.93	0	395	290	5 730			3.59	8.51
No. 5	3.81	0	631	365	7 350			2.85	6.64
No. 6	3.83	0	660	354	8 340			2.95	5.85
No. 7	0	1.36	335	335	.....			3.11	....
No. 8	0	1.37	280	280	.....			3.72	....
No. 9	1.88	1.32	515	370	8 070			2.81	6.04
No. 10	1.90	1.34	510	356	8 490			2.92	5.75
No. 11	3.86	1.36	725	369	9 570			2.82	5.10
No. 12	3.80	1.33	691	336	9 690			3.10	5.03
No. 13	1.94	0.75	536	388	8 070			2.68	6.04
No. 14	1.93	0.75	500	370	7 080			2.81	6.89
No. 15	3.83	0.74	710	376	9 060			2.77	5.38
No. 16	3.84	0.75	710	294	11 130			3.54	4.37
No. 17	0	0	625	625	.....	3675		5.88	....
No. 18	0	0	654	654	.....			5.62	....
No. 19	4.15	0	1020	750	7 260			4.90	6.71
No. 20	4.10	0	1040	779	7 140			4.72	6.83
No. 21	0	1.54	975	975	.....	4521		4.64	....
No. 22	0	1.51	935	935	.....			4.84	....
No. 23	2.07	1.51	1010	878	7 200			5.15	6.77
No. 24	2.07	1.52	1005	804	10 530			5.62	4.62
No. 25	4.10	1.50	1262	908	9 600			4.98	5.08
No. 26	4.03	1.47	1205	799	10 830			5.66	4.50
No. 27	1.98	1.48	987	863	7 200			5.24	6.77
No. 28	1.95	1.46	1011	867	8 220			5.22	5.93
No. 29	2.04	1.52	970	819	8 220			5.52	5.93
No. 30	0	0	835	835	.....			5.42	....
No. 31	1.96	1.41	979	821	8 850			5.51	5.51
No. 32	2.01	0.80	1153	998	8 640			4.53	5.64

Stiffness of Reinforced Brick Columns.—Values of the secant modulus of elasticity, E, at one-fourth the ultimate load also after repetition of a load of 34 to 57 per cent of the maximum are listed in Table VI. These values of E are comparable to similar stiffness factors for reinforced-concrete columns of similar strength. Columns with 2 per cent and 4 per cent of vertical steel without hoops exhibited moduli approximately 40 and 75 per cent higher, respectively, than the plain columns. This agrees with theoretical calculations based on equal longitudinal strain in steel and concrete. The values of E for the hooped columns containing 4 per cent of steel are noticeably lower than the theoretical relationship. The lack of stiffness in the hooped columns containing 4 per cent vertical steel made with either brick also appears to be due largely to ineffectiveness of the cores in carrying their proportion of the load.

TABLE VI.—MODULI OF ELASTICITY OF BRICK COLUMNS.

Age at Test, 4 weeks.

Column	Percentage of Reinforcement		Ultimate Strength, lb. per sq. in. (P)	Modulus of Elasticity at ½ Ultimate Load, lb. per sq. in.			Ratios	
	Longitudinal	Lateral		Column (E)	Column After Set	Prism (E_1)	E/E_1	E/E_2[a]
No. 1	0	0	983	1 410 000	....	905 000	1.56	0.93
No. 2	0	0	1090	1 610 000	1 760 000		1.78	1.07
No. 3	1.92	0	1667	1 910 000	....		2.11	1.26
No. 4	1.93	0	1580	2 070 000	....		2.29	1.42
No. 5	3.81	0	2525	2 580 000	....		2.85	1.71
No. 6	3.83	0	2640	2 380 000	2 160 000		2.63	1.58
No. 7	0	1.36	1340	1 430 000	1 370 000		1.58	0.95
No. 8	0	1.37	1120	1 110 000	940 000		1.23	0.74
No. 9	1.88	1.32	2060	1 920 000	1 860 000		2.12	1.27
No. 10	1.90	1.34	2040	1 800 000	1 650 000		1.99	1.19
No. 11	3.86	1.36	2900	2 270 000	2 240 000		2.51	1.50
No. 12	3.80	1.33	2765	2 140 000	2 110 000		2.37	1.42
No. 13	1.94	0.75	2145	1 990 000	1 900 000		2.20	1.32
No. 14	1.93	0.75	2000	2 120 000	1 980 000		2.34	1.40
No. 15	3.83	0.74	2840	2 350 000	2 220 000		2.60	1.56
No. 16	3.84	0.75	2840	1 910 000	2 110 000		2.11	1.26
No. 17	0	0	2500	3 000 000	....	2 680 000	1.12	0.96
No. 18	0	0	2615	3 500 000	3 550 000		1.31	1.10
No. 19	4.15	0	4080	4 210 000	3 800 000		1.57	1.30
No. 20	4.10	0	4160	4 370 000	3 800 000		1.63	1.35
No. 21	0	1.54	3900	3 810 000	3 920 000	3 680 000	1.04	1.17
No. 22	0	1.51	3740	3 520 000	3 680 000		0.96	1.08
No. 23	2.07	1.51	4040	4 200 000	4 150 000		1.14	1.29
No. 24	2.07	1.52	4020	2 860 000	3 050 000		0.78	0.88
No. 25	4.10	1.50	5050	3 940 000	4 060 000		1.07	1.21
No. 26	4.03	1.47	4820	3 340 000	3 630 000		0.91	1.03
No. 27	1.98	1.48	3950	4 100 000	3 730 000		1.11	1.26
No. 28	1.95	1.46	4045	3 690 000	3 350 000		1.00	1.14
No. 29	2.04	1.52	3880	3 540 000	....		0.96	1.09
No. 30	0	0	3340	3 270 000	....		0.89	1.01
No. 31	1.96	1.41	3915	3 310 000	3 600 000		0.90	1.02
No. 32	2.01	0.80	4610	4 000 000	4 060 000		1.09	1.23

[a] E_2 = Modulus of elasticity of corresponding plain column. Columns Nos. 1 to 16, E_2 = 1,510,000 lb. per sq. in.; Columns Nos. 17 to 32, E_2 = 3,250,000 lb. per sq. in.

CONCLUSIONS

1. The tests show that the effective strength of a reinforced brick masonry column equals the sum of three components, the strength of the plain masonry, the strength of the longitudinal steel at its yield point, and a less certain one due to lateral restraint offered by hoops when placed in the horizontal joints. A formula involving these components which is in reasonable agreement with the results is presented.

2. The data indicate that with very strong brick and mortar, a ratio of gross to core area of 1.7, $2\frac{1}{2}$ per cent of lateral and 7 per cent of longitudinal reinforcement based on core area, that crushing strengths of 5000 lb. per sq. in. may be obtained on gross area and 7000 lb. per sq. in. on core area alone.

3. Since the steel is the most expensive material in this type of column, economy dictates the use of strong mortars and high-strength brick together with sufficient longitudinal steel to insure proper stiffness and requisite security under eccentric loading. The addition of $\frac{3}{4}$ per cent of hoops based on gross area promoted toughness, increased the strength and eliminated sudden failures in these tests. It seems probable that for ratios of A/A_c less than 1.5, the percentage of hooping or other desirable lateral reinforcement could be reduced to 0.6 per cent.

4. Filling small cores with bats is not as effective as filling with high-strength concrete.

5. For common brick the strength of 8 by 8 by 16-in. masonry prisms was a good index of the strength of the plain columns, but for the high-strength brick the ratio of the plain column strength to the strength of the prisms was only 0.70 to 0.75.

6. With careful workmanship including filled joints and good materials, reinforced brick masonry columns should be safe under static loads with a factor of safety of 4. For columns of high-strength brick and mortar, reinforced with both longitudinal steel and hoops, a factor of safety of $3\frac{1}{2}$ is probably safe.

7. Additional column tests should be made to ascertain more clearly the action of hooping, the suitability of other types of hooping, and the efficiency of still stronger mortars in combination with high-strength brick and steel with a high yield point. Larger columns with low ratios of gross area to core area, also columns with concrete cores placed by vibration should be tested. The effects of shrinkage, duration of load on plastic flow, the durability of both brick and mortars under freezing and thawing as in foundations, and the resistance of reinforced brick columns to eccentric loads are all problems worthy of solution.

DISCUSSION

MR. INGE LYSE[1] (*presented in written form*).—An investigation on reinforced brick columns was carried out at Lehigh University.[2] The report on this investigation presented a recommended design formula which is much similar to the one proposed by Mr. Withey. The factor of safety recommended was 4.0, or the same as Mr. Withey proposes for ordinary reinforced brick columns. It is gratifying to find such complete agreement between investigations carried out at two different places, which adds weight to the conclusion that reinforced brick columns of proper design and workmanship may well be used in modern engineering construction.

MR. F. O. ANDEREGG.[3]—Last fall we built at Carnegie Institute of Technology three columns of ceramic units, 10 ft. high. The units were 3¾ by 8 in. in cross-section. A small steel rod ran through a hole in the center to facilitate assembly. The columns were tested at the age of about 40 days with plaster caps. The slenderness ratio of about 32 was quite high, and the columns suffered a bending of about an inch. Failure occurred by splitting at one end of the column at an average total load of 180,000 lb. or 6000 lb. per sq. in., which is quite high for so slender a column. It is suggested that lateral reinforcement as used by Mr. Withey is unnecessary, provided that properly designed ceramic units are employed. The tensile strength of a well-burned ceramic unit is adequate, and the wedge action of the steel hoops would be eliminated. The results obtained by Mr. Withey compare very favorably with reinforced concrete and with steel columns, especially when it is remembered that steel columns require fireproofing. Some economies doubtless are effected with reinforced brick columns over both steel and concrete with proper design and proper methods of assembly.

A MEMBER.—I notice that when the stress was maintained constant the strain continued to increase. That looks like a very great weakness, and I would appreciate further information on that observation.

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[1] Research Associate Professor of Engineering Materials, Lehigh University, Bethlehem, Pa.

[2] The results of the 33 columns included in the Lehigh University investigation were published in a detailed report entitled "Tests of Reinforced Brick Columns," *Journal*, Am. Ceramic Soc., November, 1933, p. 584; also published in condensed form in *Engineering News-Record*, March 16, 1933, p. 345, and January 4, 1934, p. 12.

[3] Consulting Specialist on Building Materials, Long Island City, N. Y.

MR. M. O. WITHEY.[1]—In reply to the observation that certain columns exhibited large increases in strain under constant stress, attention is called to Fig. 8 of the paper. These and other similar data for the columns tested show that the rate of strain decreases with time, or with the number of repetitions. For loads approximating 40 per cent of the ultimate, the increase in strain with time soon becomes a very small quantity, no larger than exhibited by other semi-plastic materials under comparable intensities of stress. As mentioned in conclusion 7, the effect of duration of load on plastic flow should be studied more thoroughly.

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[1] Professor of Mechanics, University of Wisconsin, Madison, Wis.

PROPOSED METHOD OF MAKING COMPRESSION TESTS ON PORTIONS OF CONCRETE BEAMS FROM FLEXURE TESTS

By L. H. Koenitzer[1]

Synopsis

The practice of making flexure tests upon concrete for use in pavements is now almost universally established. In addition to the modulus of rupture strength, most engineers, however, also desire to secure the compressive strength of the concrete. The Kansas Highway Commission has for several years been determining the compressive strength of the concrete on paving jobs by sending in to the main laboratory portions of the specimens broken in flexure, the ends of which are cut square by a silicon carbide saw. The prisms are then capped and tested in compression. Some states have followed a similar procedure, while other states have continued to make the standard 6 by 12-in. cylinders along with the flexure specimens. Others have drilled out cores or ground down beam ends and made compression tests.

About four years ago, it came to the attention of Prof. C. H. Scholer that in some European laboratories it was common practice to make compression tests upon portions of beams from flexure tests (as modified cubes) as described in this paper. The Road Materials Laboratory of the Engineering Experiment Station of Kansas State College, in cooperation with the Kansas Highway Commission, has been making a rather informal investigation of this method and the results are herein presented.

The compressive strength of the modified cubes is closer to the compressive strength of the standard 6 by 12-in. cylinders than is the compressive strength of prisms.

Introduction

In recent years, it is becoming accepted practice to make flexure tests on concrete used on paving projects. As yet, the engineering profession does not have enough information on flexure tests to warrant their exclusive use in the design of concrete mixtures. Highway departments that make flexure tests either make 6 by 12-in. cylinder specimens from the same batch of concrete for compression tests, or use silicon carbide saws to saw the ends of the flexure specimens off square, making prism specimens which are tested in compression.

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[1] Assistant Professor, Applied Mechanics Dept., Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

To eliminate the necessity of buying expensive sawing equipment and extra labor required in making additional specimens, the author has attempted to perfect a suggested method of compression testing on the broken flexure specimens, the results of which compare very favorably with compression tests made in the standard manner on 6 by 12-in. cylinders. This method has been checked and found satisfactory by using transparent models in photo-elastic equipment.

MATERIALS

Two mixes were used: one consisting of sand-gravel (mixed aggregate) aggregate, and the other mix consisting of fine aggregate and limestone aggregate. The materials all met the specifications for Kansas highway pavement. Ordinary portland cement was used with the sand-gravel mix and an early-strength cement was used in the coarse-aggregate mix. A water-cement ratio of 5 gal. of water per sack of cement was used for the sand-gravel mix and 5.25 gal. of water for the coarse-aggregate mix. All tests were made on typical paving mixes.

TEST METHODS

The 6 by 12-in. cylinder compression specimens were made in accordance with the A.S.T.M. Standard Methods of Making Compression Tests of Concrete (C 39).[1] The 6 by 6 by 36-in. beam specimens were made in accordance with the A.S.T.M. Tentative Laboratory Method of Making Flexure Tests of Concrete Using a Simple Beam with Center Loading (C 78 - 30 T).[2]

The beams were broken in flexure in a simple-beam testing machine,[3] loaded at the center of an 18-in. span. The specimens were broken at the ages of 4 and 10 days, which is common highway practice.

After the beam is tested in flexure in the field, one, two, or three pieces (depending upon the number of compression tests desired) are sent to the testing laboratory where a 300,000-lb. compression machine is available. The portions of the beam are capped on the sides (not the top or bottom of the beam as it is molded) with a mixture of one part cement and one part plaster of Paris so that there shall be two smooth surfaces directly opposite each other, each surface being at least 7 in. in length and the full width of the specimen. It is recommended that machined cast-iron plates be used to place the specimen on while the cap is being formed.

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[1] 1933 Book of A.S.T.M. Standards, Part II, p. 230.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 1027 (1930); also 1933 Book of A.S.T.M. Tentative Standards, p. 440.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 589 (1930).

After capping, the specimens are placed in the moist room until time of testing when they are tested, as shown in Fig. 1, in a 300,000-lb. compression machine. The specimen is placed in the machine with 6 by 6-in. bearing plates on the top and bottom directly opposite one another; thus the specimen is tested as a modified cube having the

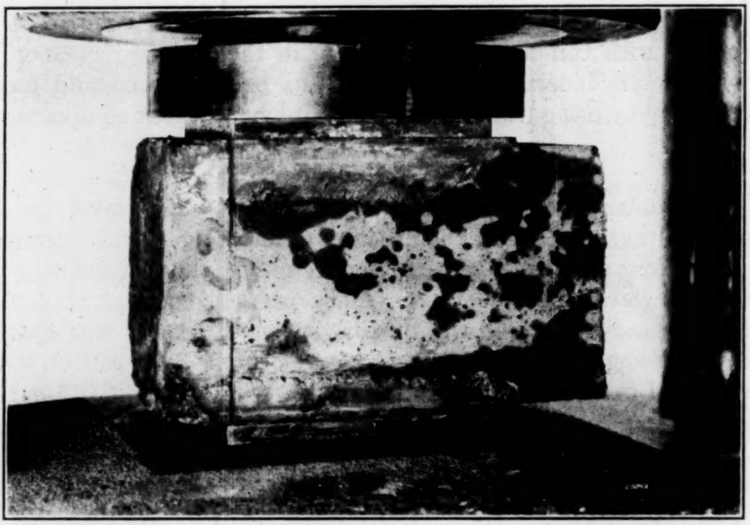

FIG. 1.—Method of Loading Modified Cube Specimen.

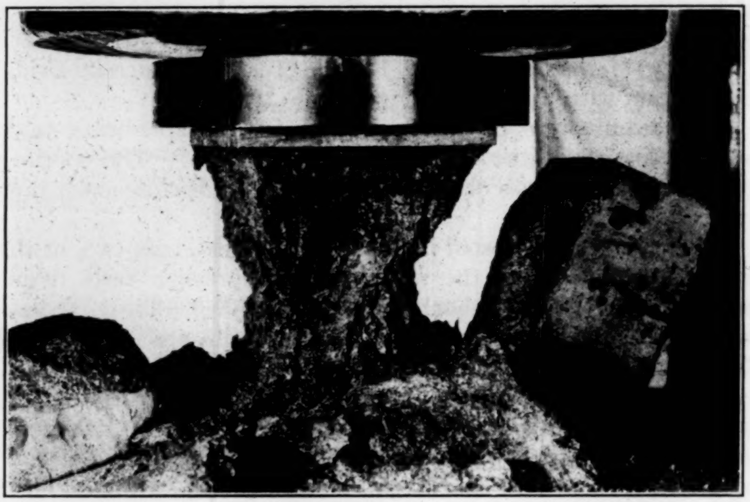

FIG. 2.—Failure of Modified Cube Specimen in Compression.

load applied over a 6 by 6-in. area, and a depth of 6 in. (if the beam originally was 6 by 6-in. in cross-section) with a short overhanging portion of the specimen on each side of the plates (Fig. 1). The length of projection beyond the plates has been from 1 to 3 in. In every case an effort has been made to have this projection symmetrical on each side. The photo-elastic analysis, however, does not indicate that this is important.

DISCUSSION OF DATA

The specimens in almost every case break up into a cone and fragments as shown in Fig. 2. In Fig. 3 are shown two sets of specimens after testing, each set having consisted originally of two 6 by 12-in. cylinders, and one 6 by 6 by 36-in. beam from a single batch of concrete. From each beam it was possible to get one prism, and two specimens tested as modified cubes. The specimens in Fig. 3 do not show as distinct a cone-break as would have been possible if the deformation had been continued.

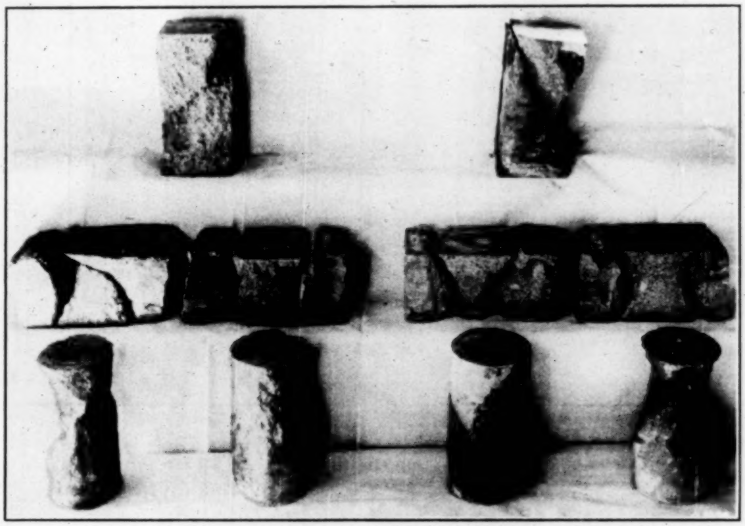

FIG. 3.—Failure of Prisms, Modified Cubes, and Cylinders Taken from the Same Batches of Concrete.

The results of Mr. Gonnerman[1] show that at the age of 28 days 6-in. cubes are 16 per cent stronger than 6 by 12-in. cylinders. This does not hold true for a comparison of the modified cube method suggested above, and 6 by 12-in. cylinders. The data in Table I

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[1] H. F. Gonnerman, "Effect of Size and Shape of Test Specimen on Compressive Strength of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 248 (1925).

TABLE I.—SAND-GRAVEL AGGREGATE CONCRETE.

Specimen	Compressive Strength, Age 28 days				
	Cylinders,[a] lb. per sq. in.	Prisms, lb. per sq. in.	Variation from Cylinders, per cent	Modified Cubes,[a] lb. per sq. in.	Variation from Cylinders, per cent
No. 1	4598	3917	−14.8	3666	−20.6
No. 2	4350	3472	−20.4	3805	−12.5
No. 5	3714	3306	−11.0	3792	+2.1
No. 6	3466	3472	+0.2	3930	+13.4
No. 9	4386	4167	−5.0	4305	−1.8
No. 12	4032	3889	−3.5	3504	−13.1
No. 13	3501	4944	+41.4	3528	+0.8
No. 16	2900	2906	−0.2	2944	+1.5
No. 17	3006	2444	−18.7	3222	+7.2
No. 20	3148	2750	−12.6	3230	+2.6
No. 21	3466	3056	−11.8	2611	−24.7
No. 24	3289	2861	−13.0	3458	+5.1
No. 25	3466	3083	−11.0	3097	−10.6
No. 28	3572	3139	−12.5	3000	−16.0
No. 29	3431	3444	+0.4	3625	+5.1
No. 37	5135	4070	−20.8	4410	−14.1
No. 38	4740	4310	−9.1	4775	+0.7
No. 39	5040	4440	−11.9	4715	−6.4
No. 40	4425	4550	+2.8	4720	+6.7
No. 41	4080	4265	+4.5	4172	+2.3
No. 42	3770	4580	+21.5	4452	+23.4
No. 43	4052	3500	−13.6	3388	−16.4
No. 44	3468	3315	−4.4	3744	+8.0
No. 45	3477	3790	+6.1	4168	+19.9
No. 46	3945	3230	−18.1	3513	−11.0
No. 47	3025	3240	+7.1	3772	+24.7
No. 48	3730	3610	−3.2	3832	+2.7
No. 49	4855	3490	−28.1	4962	+2.2
No. 50	5030	4550	−9.5	4695	−6.7
No. 51	5115	3930	−23.2	4992	−2.4
No. 52	5085	5280	+3.8	5150	+1.3
No. 53	5270	5210	−1.1	5470	+3.8
No. 54	4935	5140	+4.2	5110	+3.5
Average	4045	3797	−5.7	3993	−0.6

[a] Each value is the average of two specimens.

TABLE II.—COARSE-AGGREGATE CONCRETE.

Specimen	Compressive Strength, Age 14 days				
	Cylinders,[a] lb. per sq. in.	Prisms, lb. per sq. in.	Variation from Cylinders, per cent	Modified Cubes,[a] lb. per sq. in.	Variation from Cylinders, per cent
No. 61	4200	3420	−18.7	3735	−11.1
No. 62	3820	3310	−13.4	3695	−3.3
No. 63	3140	3000	−4.5	4345	+38.4
No. 64	3460	3810	+10.1	4080	+17.9
No. 65	3990	3440	−13.8	3860	−3.3
No. 66	4030	4190	+4.0	3570	−11.4
No. 67	3530	2860	−19.0	2950	−16.4
No. 68	3670	2920	−20.5	3200	−12.8
No. 69	3790	3250	−14.3	3350	−11.6
No. 70	4749	4220	−11.1	5117	+7.7
No. 71	4925	4705	−4.5	4967	+0.8
No. 72	4155	4092	−1.5	4466	+7.5
No. 73	4460	4149	−7.0	4775	+7.1
Average	3994	3644	−8.8	4008	+0.7

[a] Each value is the average of two specimens.

indicate that it is not necessary to apply a correction factor when comparing the compression tests on 6 by 6-in. modified cubes, and 6 by 12-in. cylinders. The average strength of the 66 modified cubes made of sand-gravel aggregate was 3993 lb. per sq. in. as compared with 4044 lb. per sq. in. for the sixty-six 6 by 12-in. cylinders.

The average strength of 33 prisms was 3795 lb. per sq. in. These averages are based upon specimens made from the same batches of concrete, and the individual comparison between specimens can be made by considering that the specimens on each line (Table I) are made from the same batch of concrete.

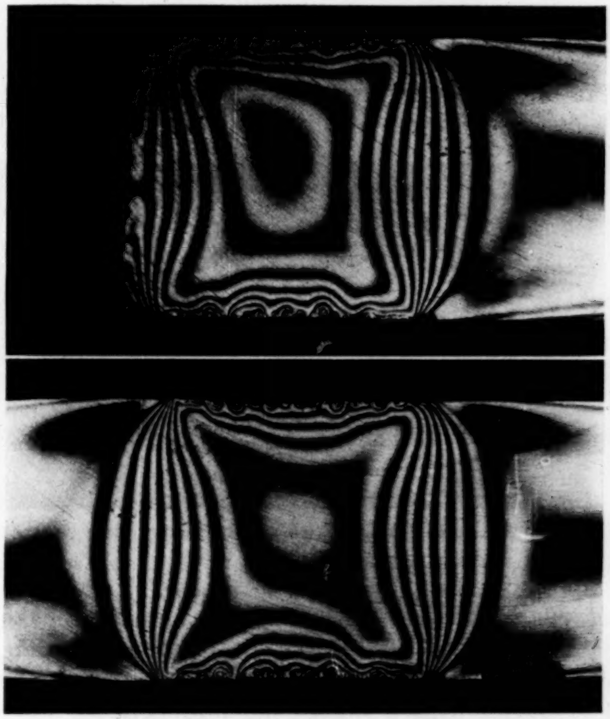

FIG. 4.—Photo-Elastic Analysis Showing Uniform Stress Distribution Regardless of the Location of Load on the Modified Cube Specimen.

For a coarse-aggregate concrete mix the average strength of twenty-six 6 by 12-in. cylinders was 3994 lb. per sq. in. (Table II), and the average strength of 26 modified cubes from the same batches of concrete was 4008 lb. per sq. in. Prisms sawed out of the same flexure specimens from which the modified cubes were taken show a strength of only 3664 lb. per sq. in., which is 8.8 per cent less than the strength of the corresponding cylinders.

The strength of the modified cubes corresponds very closely to the strength of 6 by 12-in. cylinders. The modified cube method gives much better results than the prisms when compared to the standard cylinder specimens. In fact, there is a closer relationship between the strength shown by cylindrical specimens and the modified cube specimens than is shown between the cylinders and the prisms. Tests on over 300 modified cubes which were not capped indicate that regardless of the apparent condition of the sides of the beams it is always necessary to cap both sides of the broken flexure specimen before testing in compression. Modified cubes, not capped, were 8 per cent lower in strength than the capped standard 6 by 12-in. cylinders, while capped modified cubes varied only 1.25 per cent from the standard cylinders.

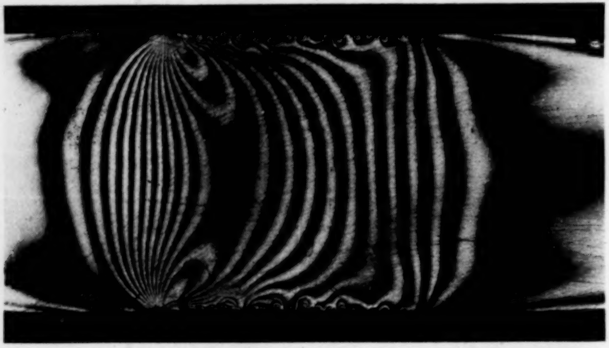

FIG. 5.—Photo-Elastic Analysis Showing Concentration of Stresses Due to Eccentric Loading.

Photo-Elastic Analysis.—A photo-elastic analysis of the modified cube method is shown in Fig. 4. It indicates that the uniform stresses[1] (indicated by the dark lines) are fairly constant regardless of whether there is only a slight overhang (as in the top of Fig. 4), or whether the load is applied at the center of the specimen with an overhang of two to three times the depth of the specimen.

The most important things brought out by the photo-elastic analysis are that the bearing faces of the specimen being tested have plane smooth caps or faces, that the spherical bearing block is in good working condition, and that the load is applied perpendicular to the capped surface of the specimen. It will be noted that in spite of the polishing done on the bakelite used in the photo-elastic apparatus, the material showed irregular stresses on both surfaces of the specimen. The result of not centering the specimen under the application of load shows an unequal distribution of stresses. In Fig. 5 is shown the high concentration of stresses on the left side on the specimen which is shown by the large number of black lines.

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[1] E. G. Coker and L. N. G. Filon, "Photo-elasticity," pp. 198–201.

SUMMARY

1. The length of projection of the ends of the beam past the bearing plates appeared to have no effect upon the strength of the modified cube.
2. The modified cube method requires less labor, less expenditure for maintaining equipment, and less equipment than is required to test specimens as prisms or cores.
3. The strengths obtained by the modified cube method are closer to the strength of the standard 6 by 12-in. cylinders than are the strengths of the prisms.
4. The photo-elastic analysis indicates that the stress distribution is essentially the same regardless of the amount of overhang on each side of the bearing plates.
5. The photo-elastic analysis indicates that it is impossible to get caps smooth enough so that there will be a uniform distribution of stresses without small concentrated stresses near the surface of the specimen.
6. The photo-elastic analysis emphasizes the importance of getting the specimen accurately centered under the load so as to prevent an uneven distribution of stresses in the specimen. Highly concentrated stresses give low values not indicative of the true strength of the specimen.
7. The adoption of the modified cube method of testing makes it practical to make compression tests on beam specimens after they have been tested in flexure, without the purchase of additional equipment.
8. It is suggested that the modified cube method of making compression tests be given further study to determine its suitability for use on highway projects so as to further the gathering of valuable data on the relationship between flexural and compressive strengths of concrete mixtures.

Acknowledgment.—The author is indebted to Prof. C. H. Scholer, Director, Kansas Highway Testing Laboratory, Manhattan, Kans., for his valuable suggestions in connection with this research project. The author is also indebted to Mr. H. Allen, Materials Engineer, Kansas State Highway Commission, for the preparation of the prism specimens.

DISCUSSION

MR. R. W. CRUM[1] (*presented in written form*).—In comparing several different methods of testing a given property of a material, one factor that was only lightly touched upon by Mr. Koenitzer is the degree of uniformity attained in the test specimens. For instance, two methods might give almost identical values for the average of a given number, but the individual results of one set might be so much more widely dispersed than the other as to make a comparison on the basis of averages very misleading. That is, if one method of test showed a much wider dispersion than another for a large enough group of specimens to constitute a statistical universe, the probable error in the average of a small sample tested by the first method would be much greater than that of the average of a similar sample tested by the more uniform method.

There are several measures of uniformity used in statistical analyses that can be applied to a group of items all of which are intended to measure the same function and all of which are entitled to equal credence. The average tells nothing of how the individual items are arranged between the maximum value at one end of the range and the minimum at the other. The measure in most common use is the "standard deviation" obtained by squaring the deviation of each individual result from the average, adding them and then extracting the square root of their average. It is sometimes expressed as a percentage of the average.

On applying this process to Table I of the paper, standard deviations were computed as follows:

Cylinders	17.81 per cent
Prisms	19.44 per cent
Modified cubes	18.30 per cent

It appears from these figures that there is little difference in uniformity in the three groups of specimens, although the modified cubes approach the cylinders more closely than do the prisms, as was also shown by the averages.

In view of the fact that these standard deviations are rather greater than would be expected if a greater number of specimens had been tested, I suggest the desirability of further observations on much larger groups so that the relative uniformity possible of attainment by the two methods of test can be more definitely determined.

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[1] Director, Highway Research Board, National Research Council, Washington, D. C.

MESSRS. H. J. GILKEY[1] AND GLENN MURPHY[1] (*by letter*).—When this paper first became available, the writers were laying out a series of compression tests on a large number of 2-in. cubes, 3 by 6-in. cylinders and 2 by 2 by 4-in. prisms (side cast) along with a few 6 by 12-in. control specimens. A number of the prisms were allocated, therefore, to tests paralleling those described in the paper. Results from these tests are tabulated below:

	7-day Tests			28-day Tests				
	Prisms, 2 by 2 by 4 in.	Prisms as Modified Cubes	Cubes, 2 in.	Prisms, 2 by 2 by 4 in.	Prisms as Modified Cubes	Cubes, 2 in.	Cylinders, 3 by 6 in.	Cylinders, 6 by 12 in.
Number of tests	12	16	24	24	8	60	8	2
Average strength, lb. per sq. in.	3035	3020	2950[a]	5570	5750	6040	5590	5280
Ratio to prism strength	1.00	1.00	0.97[a]	1.00	1.03	1.08	1.00	0.95
Max. strength any specimen, lb. per sq. in.	3370	3400	3460	6175	6200	6825	5800	5330
Min. strength any specimen, lb. per sq. in.	2500	2600	2740	5100	5325	5000	5410	5220

[a] Probably out of line on the low side. Cubes are usually somewhat stronger than prisms and cylinders of height twice the lateral dimension.

From these limited tests the following comments are ventured. Support is found for conclusions Nos. 1, 2, 3, 4, and 8 of the paper. As a check on conclusion No. 1, for half of the 24 prisms tested as cubes the modified cube was taken as the middle 2 in. of the 4-in. length and for the others, as the end 2 in. Moreover the zone of damage does not (for the mortar used) extend beyond the boundary picked for the cube. For tests in which the modified cube was taken as an end 2 in. of the prism, the other 2 in. was undamaged and gave virtually the same strength as the end tested first. Sometimes the strength of the fragment was higher rather than lower. Still more striking was the fact that the two end fragments resulting from the test when the middle 2 in. were used as the cube gave a strength which equalled that of the middle 2 in. at the original test.

It is difficult to understand why a prism tested as a modified cube should be weaker than a cube. It is common experience, as mentioned at the bottom of page 409 of the paper, that cubes are stronger than standard cylinders (our 7-day test results to the contrary notwithstanding). Thus while the evidence available seems to support conclusion No. 3, a satisfactory explanation is not yet apparent.

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The writers' specimens were of mortar $1:2\frac{3}{4}$ by weight and a water-cement ratio of 0.80 (sand dry and no allowance for absorption). Slump was 8 to 10 in.

In closing, it is especially desired to commend the author on calling attention to a step in testing technique that promises to simplify greatly the control operations in pavement and some other forms of concrete construction.

THE CHAIRMAN (*Mr. F. E. Richart*[1]).—The Chair would like to ask why the load was applied to the sides of the beam instead of to the top and bottom, as the beam was originally molded.

MR. W. E. GIBSON.[2]—It was the thought of the author that a much smoother bearing surface can be obtained by using the side of the beam, as the top of the beam is generally rounded, due to casting.

MR. L. H. KOENITZER[3] (*author's closure by letter*).—The author is indebted to Mr. R. W. Crum for his suggestions and computation of the standard deviation for the sand-gravel aggregate concrete. The author considered applying statistics to the data but questioned the application of statistical methods to a small amount of data. Applying the same statistical method to Table II of the paper, the results are as follows:

Cylinders	12.3 per cent
Prisms	15.5 per cent
Modified cubes	16.5 per cent

To find the deviation of the strength of specimens made in the field, the author selected at random two concrete paving jobs which had sent to the laboratory the broken ends of the flexure specimens. The ends were cut square on a silicon carbide[4] saw. The specimens were then capped and tested in compression as prisms, at the age of 90 days. On one project the standard deviation was 10.58 per cent for 66 specimens, and on another project the standard deviation was 11.1 per cent for 57 specimens. It is obvious that the larger the number of samples (specimens) the more significant will be the standard deviation.

Some of the data in Table I of the paper were secured from field specimens which would not be expected to fit in closely with the laboratory specimens.

The author is also indebted to Messrs. H. J. Gilkey and G. Murphy for their interest in checking the results of the paper using

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Engineer of Tests, Kansas Highway Commission, Manhattan, Kansas.

[3] Assistant Professor, Applied Mechanics Dept., Kansas State College of Agriculture and Applied Science, Manhattan, Kans.

[4] C. H. Scholer and L. H. Koenitzer, "A Study of Fourteen Brands of Standard Portland Cement, and Four Early-Strength Cements," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 590 (1930).

2-in. mortar specimens. It is very difficult to mold 2-in. specimens uniformly; hence, one would not expect a close agreement in their strength results. However, the data secured by Messrs. Gilkey and Murphy bear out the conclusions of the author.

Since the presentation of the paper at the annual meeting, the Kansas Highway Commission, under the direction of Mr. H. Allen, Engineer of Materials, and Mr. W. E. Gibson, Engineer of Tests, has made an independent study of the modified cube method in conjunction with another research problem.

TABLE I.—RESULTS OF TESTS BY KANSAS STATE HIGHWAY COMMISSION.

Batch	Compressive Strength, lb. per sq. in.			
	Cylinders, 6 by 12 in.	Prisms, 6 by 6 by 12 in.	Modified Cubes, 6 by 6 in.	Cubes, 6 by 6 by 6 in.
No. 1	4223	3790	4220	4160
No. 2	4320	3430	4080	3800
No. 3	4328	4077	4688	4320
No. 4	4594	4035	4389	3770
No. 5	5088	4250	4954	4809
No. 6	4346	4220	5229	4389
No. 7	4947	3923	4813	4333
No. 8	4187	4053	4100	4260
No. 9	4611	4466	4459	5711
No. 10	4611	4472	4325	4817
No. 11	4417	4280	3736	4792
No. 12	3781	4289	4063	4854
No. 13	4205	4230	3680	3300
No. 14	2611[a]	3953	4070	3570
No. 15	4350	4335	3930	2915
No. 16	3220	3645	3805	3545
No. 17	4210	4185	4515	4220
No. 18	3430	3640	3500	3195
No. 19	3499	3833	4583	3778
No. 20	3216	3472	3250	2556[a]
Average	4189	4029	4219	4134
Standard deviation, per cent	12.43	7.52	11.57	16.31

[a] Omitted from the results since these values varied more than 2 × standard deviation from the average.

One 6 by 6 by 36-in. beam, and two 6 by 12-in. cylinders were made out of the same batch of sand-gravel concrete. The specimens were tested as described in the paper with one exception: one portion of each beam was cut with a silicon carbide saw to form a cube 6 by 6 by 6 in. which was capped and tested in compression. The data given in the accompanying Table I are the results of the above tests. The reader should keep in mind that comparison between specimens should be made for each batch as each batch varied slightly, depending upon the variables which were being studied. However, the author believes that there is not enough variation between batches to warrant the rejection of the data and that it is possible to compare the various methods of tests for several mixes and water-cement ratios.

As shown in Table I, the 6 by 12-in. cylinder specimens had a wide range of deviation from the average, while the prisms had a much smaller standard deviation. The deviation of the strengths of the modified cubes were slightly less than for the cylinder specimens, and more than the prism specimens. The 6 by 6 by 6-in. cube specimens had the greatest deviation. The question has always been raised when concrete specimens are cut with a silicon carbide saw, as to whether the heat from sawing or the fatigue of the concrete near the sawed surface alters the results when the specimens are tested in compression. Such questionable factors do not enter in when the specimens are tested as modified cubes.

As a check on field specimens, several groups of field specimens were separated in the laboratory according to projects: one-half of the specimens selected at random were tested as prisms, the other half of the specimens were tested as modified cubes. A tabulation of the results on two projects studied is given below:

FIELD SPECIMENS

Project	Number of Specimens (each method)	Prisms		Modified Cubes	
		Average Strength, lb. per sq. in.	Standard Deviation, per cent	Average Strength, lb. per sq. in.	Standard Deviation, per cent
No. 1	13	5270	13.5	5423	12.5
No. 2	11	5594	17.9	5309	16.3

It will be noted the standard deviation of the modified cube specimens are less than the standard deviation of the prisms.

The Kansas Highway Commission at the present time is testing all of the broken flexure specimens sent in from the field (for compression tests) as modified cubes, as outlined in this paper. The adoption of this method of test will mean a saving of several thousands of dollars a year for the State of Kansas.

Engineers who have made a study of the modified cube method since the presentation of the paper, have found that most of the conclusions given in the paper were substantiated by their results and their data gave better results than the author's data which was based upon field and laboratory specimens combined.

PERMEABILITY TESTS OF 8-IN. BRICK WALLETTES[1]

By L. A. Palmer[2] and D. A. Parsons[2]

Synopsis

A total of 240 wallettes made with 10 representative mortars and 5 makes of bricks were aged for 3 months and tested for permeability by keeping a constant level of water in a shallow reservoir (1 in. in depth) on the upper surface (face) of each test specimen. Each wallette was constructed with 16 bricks (both header and stretcher bricks) and the mortar joints were approximately ½ in. in thickness. The most water-tight walls were obtained with smooth impervious bricks set dry and smooth porous bricks set wet (15 minutes total immersion). The degree of water-tightness of the wallettes was also dependent on the working properties of the mortars.

Introduction

During recent years, the problem of water transmission through masonry has received much consideration for various reasons, the more important of which are the following:

1. The recent tendency in design for thinner walls and more freely exposed masonry,
2. The increased speed of construction, and
3. An increased diversity of building units and cementing materials with too little consideration for their mutual suitability.

Although the majority of brick buildings have dry interiors, there have been numerous and annoying instances of water transmission.

An earlier investigation of the causes and possible means of prevention of efflorescence on face-brick walls[3,4] had involved a study of the problem of water transmission. It was realized at the outset that there were two possible general causes of wet wall interiors.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associates for the Research Committee of the Mortars and Masonry Research Fellowship supported by the American Face Brick Assn., the National Lime Assn., the Portland Cement Assn., and a group of manufacturers of masonry cements.

[3] L. A. Palmer, "Cause and Prevention of Kiln and Dry-House Scum and of Efflorescence on Face Brick Walls," National Bureau of Standards *Technologic Paper No. 370* (1928).

[4] "Wet Walls and Efflorescence," publication by the American Face Brick Assn., A.I.A. File No. 5m.

One of these is the matter of wrong design, faulty construction, poor workmanship and lack of proper maintenance. The other is the possibility of using an unsuitable combination of brick and mortar. It is the latter that has received considerable laboratory study[1,2,3,4,5] culminating in the present investigation.

It is pertinent to review certain findings[5] relative to the effects of rates of absorption of bricks and "water-retaining capacities"[6] of mortars on the extent of bond (adhesion) because of the probable bearing of the bond on the permeability of walls. The data indicated that it was particularly difficult under certain conditions to obtain a good extent of adhesion of mortar to brick. The data further indicated that, in the matter of getting a good extent of bond, rate of absorption of bricks and the water-retaining capacity of mortars were very important considerations.

In general, mortars of medium or high water-retaining capacity tended to give a better extent of bond, particularly with bricks of medium to high rates of absorption, than mortars deficient in this property. Also, bricks having rates of absorption not greater than 40 g. of water per minute per 30 sq. in. of brick surface produced a better extent of bond with all mortars, whether of low, medium or high water-retaining capacity, than bricks of higher rates of absorption.

The desirability of some practical test of walls for water-tightness was early recognized although there is considerable disagreement as to what really is a practical test and the procedure herein described is not suggested as the best example of practicality.

The purpose of this study was to correlate those properties of bricks and mortars which control the bond, with the permeability of small test walls. Although this work was discontinued before a desirable number of tests could be made, the data, obtained for 240 wallettes with five makes of brick and ten mortars, to indicate such a correlation.

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[1] L. A. Palmer and J. V. Hall, Jr., "Durability and Strength of Bond Between Mortar and Bricks," National Bureau of Standards *Journal of Research*, Vol. 6, p. 473 (1931).

[2] L. A. Palmer, "Volume Changes in Brick Masonry Materials," National Bureau of Standards *Journal of Research*, Vol. 6, p. 1006 (1931).

[3] L. A. Palmer, "Water Penetration Through Brick Mortar Assemblages," *Journal*, Clay Products Inst. of Am., Vol. 1, No. 1, p. 19 (1931).

[4] L. A. Palmer and D. A. Parsons, "The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. XXXV, No. 18, p. 18 (1932).

[5] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

[6] Water-retaining capacity is the measurable resistance of a mortar to water loss when on a porous base. It is considered a measure of workability, and for the purpose of this paper the percentage of flow of the freshly mixed mortar on a flow table after one minute suction on a "standard" porous base is considered an index of water-retaining capacity.

TABLE I.—BRICK USED IN MAKING WALLETTES FOR PERMEABILITY TESTS.

Brick	Raw Materials	Molding Process	Water Absorbed[a] by Dry Brick, g.	Period of Total Immersion, min.	Water Absorbed[a] After Total Immersion, g.	Surface Texture
No .1.....	Surface clay.....	Dry-press...........	117.3	15	3.2	Mechanically smooth on all sides. No cracks.
No. 2.....	Shale...........	Stiff-mud, end cut ...	22.9	15	2.1	Very rough on all sides except one edge. No cracks.
No. 3.....	Shale...........	Stiff-mud, side cut...	10.4	not determined		Moderately rough on flat sides, faces and header ends smooth and flashed in some instances. No cracks.
No. 5.....	Fire clay.........	Stiff-mud, side cut...	1.5	not determined		Flat sides smoother than most stiff-mud bricks. Ends and sides smooth and glassy. No cracks.
No. 6.....	Surface clay.....	Stiff-mud, side cut...	71.8	½	51.1	Smooth for stiff-mud bricks. Sizable cracks in nearly all specimens.

[a] Tests for absorption by partial immersion, flatside down in ⅛ in. of water for 1 min.

TABLE II.—MORTARS USED IN THE TEST.

Mortar Designation	Cementing Materials Used	Description of Cementing Materials	Proportions						Water-Retaining Capacity
			By Bulk Volume[b]			By Weight[c]			
			Cement	Lime	Sand	Cement	Lime	Sand	
B I.......	Masonry cement No. 1.......	Modified natural cement[a]..........	1	0	3	1	0	4.62	Medium
B III......	Masonry cement No. 3........	Hydraulic lime......	1	0	3	1	0	6.14	Low
B IV......	Masonry cement No. 4........	Slag and hydrated lime.............	1	0	3	1	0	4.48	Low
B VI......	Masonry cement No. 6........	Modified natural cement[a]..........	1	0	3	1	0	5.09	Low to medium
B XI......	Masonry cement No. 11.......	Modified natural cement[a]..........	1	0	3	1	0	5.18	High
C II......	Lime No. 2.....	Dolomitic hydrated lime.............	0	1	3	0	1	10.52	Low to medium
XRa......	Portland cement No. 1 and lime No. 1...	High calcium quicklime. Typical gray portland cement..	1	1	6	1	0.42 dry hydrate[d]	6.88	Medium
XVb......	Portland cement No. 1 and lime No. 4...	Dolomitic quicklime. Typical gray portland cement......	1	2	9	1	0.95 dry hydrate[d]	10.31	High
XRc......	Portland cement No. 1 and lime No. 1...	High calcium quicklime. Typical gray portland cement..	1	3	12	1	1.27 dry hydrate[d]	13.75	High
XTd......	Portland cement No. 1 and lime No. 3 ...	Hydrated high calcium lime. Typical gray portland cement...........	1	0.15	3	1	0.06	3.44	Low

[a] Contained metallic stearate.

[b] Dry rodded volumes of sand and of the dry cementing materials. For the lime putties, the volumes are of the putties as used.

[c] The weight proportions were based on the dry weights of sand and of the dry cementing materials. For mortars containing lime putty, the weight proportion of lime was based on the dry weight of the putty after drying for 24 hr. at 115 C.

[d] The amount of dry hydrate per unit weight of freshly slaked putty was obtained by drying small samples of the putty for 24 hr. in an oven at about 115 C.

MATERIALS

Cementing Materials:

The limes and cements selected for this work have been described.[1] The four limes (Nos. 1 to 4, inclusive), portland cement No. 1 and masonry cements Nos. 1, 3, 4, 6 and 11 of the listed cementing materials were selected as representative of fifty mortars studied.

Sand:

Potomac River sand was used in all tests, its screen analysis being as follows:

Passing U. S. standard sieve No. 8 and retained on sieve No. 16, per cent	7
Passing U. S. standard sieve No. 16 and retained on sieve No. 30, per cent	24
Passing U. S. standard sieve No. 30 and retained on sieve No. 50, per cent	50
Passing U. S. standard sieve No. 50 and retained on sieve No. 100, per cent	16
Passing U. S. standard sieve No. 100, per cent	3
Total	100

Brick:

Five of the six types of brick previously described[2] were included in this study. The properties of these brick are described in Table I. Most of the individual bricks of No. 6 type were badly cracked. The cracks were deep, extending in some instances to the center.

TEST PROCEDURE

Proportioning:

The weights per cubic foot of the sand and cementing materials and the method followed in getting these data are published.[3] The amount of mixing water used with the mortars was that calculated (from previous data) as producing normal flow (100 to 110) on the flow table.[4] A description of the 10 mortars used in this study is given in Table II. Limes Nos. 2 and 4 were dolomitic and Nos. 1 and 3 high calcium. Nos. 1 and 4 were pulverized quicklimes and Nos. 2 and 3 were hydrates.

Making the Test Specimens:

The design of the test wallette is illustrated by Fig. 1. The joints, both vertical and horizontal, were approximately $\frac{1}{2}$ in. thick. Every effort was made to have all joints full and flush with the wall. Work was done as rapidly as possible so as to get good contact between the highly porous bricks and mortars of low water-retaining capacity and to minimize loss of water by segregation in such mortars when remaining uncovered on impervious bricks.

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[1] See Table I in paper by L. A. Palmer and D. A. Parsons, "The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. XXXV, No. 18, p. 18 (1932).

[2] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

[3] L. A. Palmer and D. A. Parsons, "The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. XXXV, No. 18, p. 18 (1932).

[4] According to the method described in Section 21 (*c*) of the Tentative Specifications and Tests for Masonry Cement (A.S.T.M. Designation: C 91 – 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 698 (1932); also 1933 Book of A.S.T.M. Tentative Standards, p. 295.

The procedure was one not usually followed by masons. Only an amount of mortar sufficient to bed a single brick was spread at one time, laying one brick at a time in a course of four bricks. Had the procedure been to spread mortar over the whole 4-brick course before laying the next, it would have been impossible to obtain adhesion between mortars of low water-retaining capacity and fast-absorbing bricks.

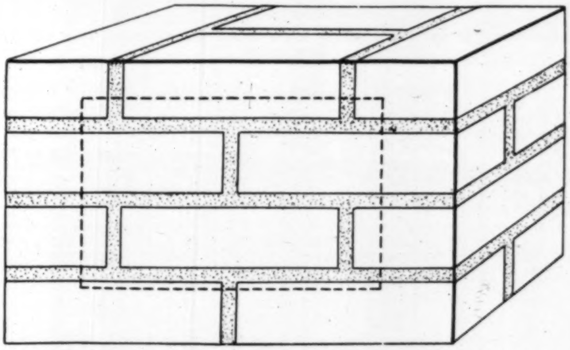

FIG. 1.—Test Wallette in the Position as Built. The dashed line marks the area covered by water during the test.

Bricks Nos. 3 and 5 were set dry in all cases. Bricks Nos. 1 and 2 were set dry and also after having been wetted by total immersion in water for 15 min. Brick No. 6 was set dry and after total immersion for one-half minute. The number of wallettes with each of the bricks Nos. 1, 2 and 6 set dry was the same as the number made with these bricks wetted, being three in each case with each brick and mortar combination. There was a total of 240 wallettes.

Curing Conditions:

All test wallettes were made and stored in a damp, unheated basement room, and tested when three months old. The temperature was never as low as freezing and except for the first month never above 65 F. (18 C.). During the last two months of the aging period the temperature was usually within the limits, 35 to 50 F. (1.7 to 10 C.). This may have been unfavorable for the development of good strength with some of the hydraulic cementing materials, but there are no available data indicating that the large number of instances of water transmission through brick walls can be explained on the basis of insufficient strength either in mortars or bricks.

Permeability Tests:

To make the test the wallette was so turned that the vertical face marked by the dashed line (Fig. 1) was uppermost and in a horizontal position, and elevated so that the bottom surface could be observed and the leakage collected for measurement.

The dashed line indicates the position of a 1⅜ by 7 by 11-in. strap iron frame sealed to the specimen by either paraffin or caulking compounds (paraffin being more suitable for rough-surfaced bricks).

TABLE III.—PERMEABILITY OF TEST WALLETTES. (ALL BRICK AND MORTAR COMBINATIONS.)

Each value is the average maximum rate recorded to the nearest half milliliter of water transmitted through 3 specimens.

Mortar	Flow After 1 Minute Suction on Standard Porous Base,[a] per cent	Brick No. 1		Brick No. 2		Brick No. 3	Brick No. 5	Brick No. 6		Average for Each Mortar, ml. per min.
		Set Wet, ml. per min.	Set Dry, ml. per min.	Set Wet, ml. per min.	Set Dry, ml. per min.	Set Dry, ml. per min.	Set Dry, ml. per min.	Partially Wetted When Set, ml. per min.	Set Dry, ml. per min.	
B I	74	2.5	77.5	39.0	217.0	24.0	0.0	322.0	185.0	108.5
B XI	87	0.0	95.0	44.0	60.5	48.5	4.0	304.0	163.0	90.0
B VI	61	7.5	94.0	15.0	60.5	21.0	0.0	205.0	289.0	86.5
XRc	91	5.5	128.0	26.5	149.0	23.0	0.0	131.0	201.0	83.0
XVb	93	40.0	98.0	40.0	132.0	6.0	0.0	155.0	400.0	109.0
XRa	75	177.0	386.0	43.0	90.0	27.0	0.0	259.0	241.0	153.0
C II	68	6.0	178.0	36.0	178.0	61.0	1.0	470.0	575.0	188.0
B III	41	10.0	317.0	97.0	192.0	33.5	0.0	537.0	710.0	237.0
B IV	48	13.0	626.0	138.0	150.0	5.5	0.0	976.0	759.0	333.5
XTd	54	7.5	402.0	226.0	226.0	165.0	0.0	608.0	854.0	311.0
Average for each brick		27.0	240.0	70.5	146.0	41.5	0.5	397.0	438.0	

[a] These values have been referred to (see footnote 4 on page 420) as approximately indicative of the relative resistances of the freshly mixed mortars to water loss when on a standard porous base.

Water was maintained within the frame at a depth of approximately 1 in. during the test.

Leaks sometimes occurred through the sides and ends of the specimens but more often through the bottom (side opposite the dotted rectangle, Fig. 1). Those leaks above the middle joint of the specimen, in its position when tested, were checked with caulking compounds. Any other leaks were considered as a part of the result.

The number of leaks, the elapsed time before they appeared, their location, and the amounts of water transmitted per minute were recorded. The duration of the test depended upon the rate of leakage. In a few instances water streamed through at such a rate that two or three liters were collected every two minutes. With other specimens, the rate was so slow that measurements were taken hourly. Usually measurements were taken for 5 and 10-min. intervals.

RESULTS

Rate of Water Penetration:

It was observed, when water penetrated through the test wall, that the initial rate of penetration was sometimes greater and sometimes less than the subsequent rate. The test was discontinued as soon as repeated measurements of collections of water indicated that the maximum rate had been determined, and any indication of a tendency to hold and not transmit water was checked by continuing the test over a period of from 6 to 7 hr. With wallettes built with porous bricks Nos. 1 and 6 the initial rate was usually less than the

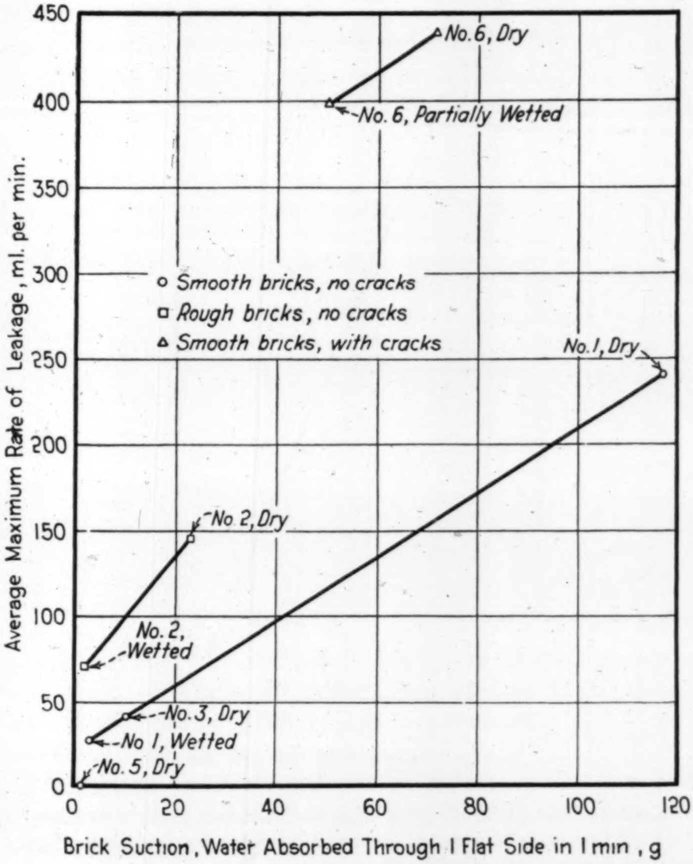

FIG. 2.—Effect of Properties of Bricks on the Maximum Rate of Leakage. **Best results were obtained with smooth and impervious bricks and also with smooth, porous bricks when the latter were wetted before laying them.**

maximum and this may have been due to the fact that a part of the water was absorbed and retained by the bricks until they gradually became saturated.

The data of Table III indicate that although the maximum rate fo water transmission varied both with the brick and mortar used, it was affected relatively more by the brick than by the mortar. The best record, that obtained with brick No. 5, smooth and very impervious, is of especial interest since it is a type of unit that has been widely associated with leaking buildings. Since it is incapable of absorbing any appreciable amount of water, it is obvious that water entering through joints left unfilled by the mason in walls of this brick must either be absorbed by the more porous back-up materials, descend between the back of the wall and the furring, or else penetrate

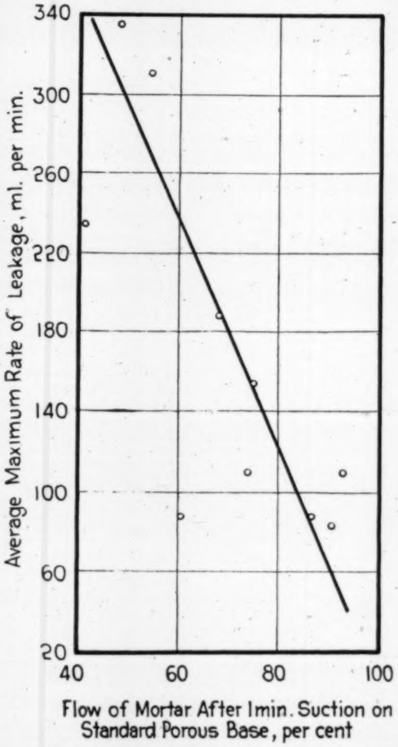

FIG. 3.—Leakage-Flow Curve Showing That the Maximum Rate of Leakage Tended to Be Highest with Mortars of Lowest Flow After Suction for a Minute on a Standard Porous Base and *Vice Versa.*

into the interior of the building. However, the results obtained with this brick when the joints are well filled may be comparatively very satisfactory, as the data of Table III indicate. This is in good agreement with results obtained by others [1,2] with this type of brick.

Relatively good results (Table III) were obtained also with brick No. 1 set wet and with brick No. 3 set dry. Poorest results were obtained with brick No. 6. Specimens of this make of brick were usually badly cracked and it is believed that the poor results in this case were in a large measure due to this condition. In general, considering all of the bricks as set dry, the rate of leakage was directly proportional to the brick suction.

TABLE IV.—AVERAGE MAXIMUM RATE OF WATER TRANSMISSION OBTAINED WITH DIFFERENT MORTARS AND ROUGH-SURFACED BRICK NO. 2 SET WET (15 MIN. TOTAL IMMERSION).

MORTAR	TROWELING PROPERTIES	AVERAGE MAXIMUM RATE OF WATER TRANSMISSION THROUGH TEST WALLETTES ML. PER MIN. AVERAGES FOR 3 SPECIMENS
B I	Excellent	39
B XI	Excellent	44
XVb	Excellent	40
XRc	Superior	26
B VI	Superior	15
XRa	Good	43
C II	Fair	36
B III	Poor	97
B IV	Very poor	138
XTd	Very poor	226 (very harsh working)

The averages for each brick, Table III are plotted in Fig. 2 against the average brick suction, Table I. In Fig. 3 the data of the second column of Table III are plotted against those of the last column. It is seen that the average maximum rate of water transmission through the test wallettes decreased as the flow of the mortars (second column of Table III) increased. However the data of Table III show that this trend did not exist with brick No. 1 set wet and bricks Nos. 3 and 5 set dry.

The flow after suction (indicative of water-retaining capacity) of a mortar may be a good index to its practical workability under the trowel. There are, however, other possible factors, one of which would be indicated by the extent to which a mortar tends to fill the small interstices on rough-textured brick surfaces, rather than "bridge across" such areas. For example, mortars C II and B VI were more

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[1] "Impervious Brick Masonry," publication by the Alton Brick Co., St. Louis, Mo. (1933).

[2] "Leaky Brick Walls and How to Prevent Them," publication by the Louisville Cement Co., Louisville, Ky., January, 1931.

plastic or workable than their flow values after suction indicated and in Table IV it is seen that relatively good results were obtained with these mortars and rough-surfaced brick No. 2. The rate of water transmission through the test wallettes was highest with the harsh-working mortars, B III, B IV and XTd.

Previous work[1] with these same 10 mortars indicated that the permeability of mortar specimens cast in metal molds, and aged for 3 months varied almost directly as the water-retaining capacity of the freshly mixed mortars, the permeability of mortar XTd being the lowest. The relatively rapid passage of water through the test wallettes made with mortars B III, B IV and XTd was due to the prevalence of unbonded areas, evident from inspection of the broken wallettes.

TABLE V.—LOCATION AND NUMBER OF RECORDED LEAKS.

Legend: *v–b* = leaks located at the juncture of vertical and horizontal (bed) joints.
b = leaks from bed joint.
v = leaks from vertical joint.
s = leaks from side (below middle section) of specimens.
c = leaks from end (below mid-section) of specimens.

	Number of Leaks With All 10 Mortars					
	v–b	*b*	*v*	*s*	*c*	Total
Brick No. 1 laid wet	19	2	0	3	0	24
Brick No. 1 laid dry	81	7	4	7	1	100
Brick No. 2 laid wet	61	6	6	10	1	84
Brick No. 2 laid dry	108	16	14	11	6	155
Brick No. 3 laid dry	49	8	2	7	2	68
Brick No. 5 laid dry	2	3	0	0	0	5
Brick No. 6 laid after partial wetting	39	6	3	6	1	55
Brick No. 6 laid dry	53	15	4	2	2	76
Total	412	63	33	46	13	567 (grand total)
Percentage of grand total	73	11	6	8	2	100

Location and Number of Leaks:

It is likely that the number of leaks per wallette is a consideration of less importance than the actual quantity of water transmitted through it in unit time. Usually the number of leaks was relatively large when the rate of water transmission was relatively high. Sometimes it was very difficult to determine the number, especially when water permeated header bricks of high absorption rate and dripped off their lower surfaces. In the latter case the water dropping from one header brick was considered as one leak.

Most of the leaks were at the junctures of bricks and mortar joints and particularly at the junctures of vertical and bed joints

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[1] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

(with reference to the position of the wallettes when made), as indicated in Table V.

DISCUSSION

Discussion as to what may be considered "normal" exposure of a wall to a wind-driven rain is beyond the purpose and scope of this paper. The opinion is offered that although the tests were much more severe than the conditions to which brick walls are normally subjected, they were more easily controlled and standardized than tests by hosing, and they also afforded data for comparative numerical values.

An accurate interpretation of these data is not possible without some means of proper comparison of the method of test with conditions obtaining in walls of buildings subjected to heavy, wind-driven rains. No attempt is made or proposed here to find such a means of comparison. The method of test followed was an arbitrary one and provided comparable data with different brick and mortar combinations. It was proposed to subject the test wallettes to an exposure which could at least be controlled and standardized even though it would be more accelerated and severe than "normal wall exposure."

Some may possibly consider that water-tightness in walls of buildings can be realized only by having conditions such that, were it possible to test the wall after the manner described herein, no water penetrates through the wall during an arbitrarily designated period of time. Although it is chiefly a matter of judgment, the opinion is offered that owing to the severity of the tests and the fact that there was no furring, plaster, etc., at the backs of the test wallettes, it is likely that the specimens which leaked only moderately would, for all practical purposes, be satisfactory from the standpoint of water-tightness. It is obviously impracticable to designate an arbitrary limiting value in rate of water transmission and state that for all practical purposes a water-tight wall would be obtained if the rate was below this value. About the only reasonable assumption that can be made is that those properties of bricks and mortar which were apparently associated with relatively slow rates of water transmission are desirable properties in any wall of brick masonry. These properties for bricks were smoothness and evenness and a very low rate of absorption when laid, the latter condition being obtained either by using a very dense, impervious brick or by wetting a porous one prior to laying it in a wall. In so far as mortars are concerned, the data indicate that high water-retaining capacity of the freshly mixed mortars diminished the rate of water transmission through the wallettes constructed of dry porous brick or rough-textured brick.

From the standpoint of bond strength alone a rough surface may be very desirable, but it is likely such a unit when used with mortars of poor workability may produce small unbonded areas which permit moisture to permeate between the mortar and brick surface. With more workable mortars, this condition is improved, as the data of Table IV indicate.

SUMMARY AND CONCLUSIONS

From the data, it is believed that the following conclusions are warranted:

1. The relative water-tightness of the wallettes depended on the properties of the bricks and mortars, the most important being the water-retaining capacities of the mortars and the absorption rates of the bricks.

2. In the tests as conducted, the water-tightness of the test wallettes was controlled more by the brick than by the mortar properties.

It is believed that there is a reasonable doubt as to this being the case in walls of buildings wherein the joints are often only partially filled. When the joints are not completely filled with mortar, water is more apt to penetrate into the interior of the building if the bricks are very impervious than would be the case were the bricks porous. Also, the use of a workable mortar tends to increase the filling of vertical joints, thus in actual construction, mortar properties may be of more relative importance than the data of this paper indicate.

3. With all joints well filled, the most water-tight wallettes, considering all mortars, were obtained with smooth, impervious bricks set dry and with smooth, porous bricks set wet.

4. From the standpoint of water-tightness, cracks in bricks are very undesirable and accelerate the rate of water transmission through the wall.

5. The wallettes made with porous bricks set dry were more water-tight with the mortars of high than with the mortars of low water-retaining capacity. It was evident from visual inspection of specimens broken after tests, that the extent of bond with porous bricks set dry increased as the water-retaining capacity of the mortars increased.

6. With rough-surfaced bricks set wet, the wallettes made with the relatively workable mortars were more watertight than those made with the other mortars.

7. Seventy-three per cent of the total number of leaks in the 240

test wallettes appeared at the junctures of the vertical and horizontal mortar joints.

8. The rate of water transmission through the wallettes made of porous bricks increased as the bricks became saturated during the tests. A wall of saturated porous bricks is similar to one of impervious bricks in that neither wall retains the water that enters it.

9. The rate of water transmission through the test wallettes was independent of the permeabilities of the mortars used. This was apparently due to the fact that in all cases the points most accessible to the entrance of water were at the junctures of bricks and mortars.

10. The results of tests with brick No. 1 set wet are in good agreement with previous results obtained with a similar type (dry-press) of brick also set wet. The previously obtained data are published in Vol. 1, No. 1, September, 1931, issue of the *Journal* of the Clay Products Institute.

RECOMMENDED PRACTICE

For the benefit of users of unit masonry materials the following points are mentioned which should be of help in getting water-tight walls.

1. All vertical joints should be well filled.

2. To aid in the filling of vertical joints a workable mortar is very necessary.

3. The consideration of next order of importance is to wet very rapidly absorbing bricks immediately before laying them. This procedure is of prime importance if the mortar lacks workability.

4. The use of bricks that are cracked, have noticeable surface irregularities, are noticeably lacking in uniformity of shape and size, or are coated with dust, soot, etc., should be avoided.

5. The mortar should have relatively low volume changes subsequent to hardening. If the extent of bond is poor, such volume changes may be very damaging and cause open cracks between the brick and mortar joints.

DISCUSSION

MR. C. C. CONNOR[1] (*presented in written form*).—The paper by Messrs. Palmer and Parsons together with their other valuable contributions to the subject of water penetration through brick masonry form an excellent background of fundamental knowledge of the properties of brick and mortar.

In addition to the conclusions given in this paper, the data apparently furnish valuable indications regarding the variation in the relative importance of certain factors known to affect the extent and durability of bond. A change in the value of one factor appears to have been reflected by a change in the amount of influence exerted by another. Part of the data and the conclusions apparently are at variance with those in my paper "Resultant Separation Cracking Between Various Mortars and Brick in Existing Brick Structures."[2] The measurements of rate of penetration and length of visible separation cracks should have given fairly parallel indications and it should prove enlightening to compare these discordant results and conclusions. It is possible that some of the methods of test used by Messrs. Palmer and Parsons may have caused the differences. The evidence regarding the variations in influence of the different factors will also be examined.

In considering the data, two points concerning the manner in which the wallettes were built and stored should be kept clearly in mind:

1. Mortar sufficient for one brick only was placed at a time, and the brick laid immediately upon it.

2. The wallettes were stored indoors and not subjected to ordinary weather conditions,which protected them from the destructive effects of extremes in temperature, and wetting and drying.

This procedure produced conditions materially different from those ordinarily obtaining in the field and may have influenced the results.

In order to compare the data in this paper with the field data presented in my paper on separation cracking, I have attempted to classify the bricks tested by Messrs. Palmer and Parsons in the same divisions that were used in presenting the field data. Bricks Nos. 1 and 2, set wet, and brick No. 5, set dry, can be safely classed as having very low rates of absorption. Bricks Nos. 1 and 6, set dry, and brick No. 6, partially wetted, unquestionably have very high rates. Unfortunately, there are insufficient data positively to identify bricks Nos. 2 and 3, set dry, with my field classifications. Very limited data, however, indicates the probability that brick No. 3 belongs in the low classification, and brick No. 2, in the high.

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[1] New Jersey Bell Telephone Co., Newark, N. J.

[2] See p. 454.

It is indicated in this paper that the best results with all mortars were obtained with bricks having very low rates of absorption. This is at variance with the data obtained in the field. The time element in laying the brick for the wallettes appears to furnish the explanation for this difference. The placing of enough mortar for one brick only, and the immediate laying of the top brick, is much different from actual practice in the field, where the mortar is spread over four or five bricks at a time and the bricks for the next course then laid. In a previous investigation, Messrs. Palmer and Parsons[1] obtained poor extent of bond with 5-brick test units where bricks wetted until they had very low rates of absorption were used with certain mortars having low water-retaining capacities. They concluded that this was likely caused by the fact that the water in such mortars tended to separate out from the solid material while it remained on the brick and prior to the pressing down of the next higher brick. The speed used in laying the bricks in the wallettes probably hindered this segregation and prevented the formation of a film of water at the bond plane where bricks of very low rates of absorption were used. In the field, ample time is allowed for the segregation of the water in most mortars, and the film thus formed is probably the major cause for the apparent tendency of very impervious bricks to increase the separation cracking with certain mortars. I am convinced that the discrepancy between the results obtained in the two investigations with such brick is due solely to the manner in which the wallettes were constructed.

I believe that the method used in building the wallettes tended to minimize the influence of the water-retaining capacity and the water-carrying capacity of the mortars with bricks having very low rates of absorption. The results obtained with brick No. 1, set wet, and No. 5, set dry, indicate this as these properties apparently had little or no effect. The rates of penetration obtained with brick No. 2, set wet, apparently were influenced by some other factor. I feel that it would be very unsafe to conclude from the evidence presented in this paper that good results could be expected in the field from combinations of bricks of very low rates of absorption and mortars having low water-retaining capacities. I also feel that there is no dependable evidence that high water-retaining capacity mortars would give satisfactory results with such bricks, although improvement is theoretically indicated.

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[1] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

The values with brick No. 2, set wet, were higher than with No. 1, set wet, which had a slightly higher rate of absorption. Obviously the difference was not caused by the suction of the brick. The influence of the rough surface of brick No. 2, as indicated by Messrs. Palmer and Parsons, appears to be a logical explanation. This is in apparent contradiction of the results obtained in the field, but the contradiction is not necessarily real. The indications of the field data were that with the more plastic mortars studied, rough surfaced brick produced less cracking than bricks having smooth surfaces. The rough surfaces possibly produced keys with the mortar and helped resist the destructive forces of differential volume change. The wallettes were protected from the extremes of volume change.

Bricks Nos. 1 and 6, set dry, and No. 6, partially wetted, all of which had very high rates of absorption, gave extremely high rates of penetration through the wallettes. It was with this class of brick that the water-retaining capacities of the mortars apparently exerted a predominating influence. This was indicated by the tremendous variation in values obtained, depending in general upon the water-retaining capacities of the mortars.

The inclusion of these values for rates of penetration in an average for each mortar with all bricks so overwhelmed the values obtained with the bricks of very low, low and high rates of absorption, that the data from these latter bricks could have been absolutely contradictory, and the resultant averages would still have indicated the controlling influence of the water-retaining capacities of the mortars. These averages gave useful information but I feel they obscured another property of mortar because they were so heavily weighted by the results from a single classification of brick. There is hardly any room for doubt of the extreme importance of the water-retaining capacity of mortar, but there are indications that it and another property of mortar exert varying influences on the extent of bond and that the proportional influence of these properties changes with changing values for the rate of absorption of brick.

In conclusion No. 1, of this paper, the capacity of a mortar to form an adequate bond layer has not been noted as being especially important. Voss[1] has demonstrated the existence of the bond layer and has indicated its importance and the probability that the formation of the bond layer is promoted by considerable amounts of lime in mortars. The field data apparently confirmed these indications where bricks of low or moderate rates of absorption were used. Inasmuch as the early stiffening of a mortar apparently tends to prevent the formation of the bond layer, its most evident influence would be looked for in combinations of the mortars studied with brick No. 3, set dry, which had a low rate of absorption. In these combinations, the shortness of time between the placing of the mortar and the setting of the brick probably prevented the formation of a film of water, but brick No. 3 had a rate of absorption sufficiently high to remove any film that might have formed and yet not high enough to cause a mortar of low water-retaining capacity to stiffen quickly. Unfortunately, there has been nothing published regarding the bond-layer-forming properties of the masonry cements included in this study. If the indications of Voss[1] and the field data are correct, however, masonry cement B IV has the property of forming an adequate bond layer because of its hydrated lime content. It has been demonstrated by Voss[1] that mortars XRa and XRb form adequate bond layers and, on the basis of lime content, XRc and C II should also have this property. Mortar XTd, however, has this property only in a very limited degree.

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[1] W. C. Voss, "Permeability of Brick Masonry Walls—an Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

In examining the data obtained with brick No. 3, it is evident that the mortars known to contain considerable amounts of lime generally produced low values, while mortar XTd produced the highest. The very low result produced by masonry cement B IV, in spite of its low water-retaining capacity, indicates the influence of some other property than noted in the conclusions, and the most probable explanation is its indicated capacity to produce a good bond layer. The value obtained from mortar C II was not in entire accordance with this explanation. The rates of penetration with brick No. 3, however, appear to be more in agreement with the indicated capacities of the mortars to form adequate bond layers where that can be judged, than with their water-retaining capacities. The field data partially confirm this since with bricks of low rates of absorption much better results were obtained from mortars containing large percentages of lime than from other mortars having virtually equal water-retaining capacities.

The data in this paper apparently furnish additional evidence concerning the range of the rate of absorption of bricks within which satisfactory results in the field might be expected. In a previous paper, Messrs. Palmer and Parsons[2] concluded that from the standpoint of bond durability, and considering the mortars studied, best results may be obtained generally by keeping the rate of absorption of bricks below the value of 40 g. of water absorbed in one minute. They also concluded that maximum bond strengths were obtained with bricks having a rate of absorption of approximately 20 g. in one minute, although this did not imply that the maximum extent of bond was so obtained. Concerning these conclusions, it is of interest to compare the results of this investigation with brick No. 2, set wet, and Nos. 2 and 3, set dry. The method of laying apparently would tend to reduce the rates of penetration with brick No. 2, set wet, more than the others, but in spite of this, better results were generally obtained with brick No. 3, set dry. Brick No. 2, set dry, produced higher penetration values than either of the other two. Its rate of absorption was 22.9 g. in one minute, which approaches the optimum rate previously given by Messrs. Palmer and Parsons[1] for strength of bond. As before stated, very limited data indicate the probability that brick No. 2, set dry, belongs in the high class by rate of absorption, which, in the field, apparently tended to increase the amount of separation cracking. The rough surface of this brick is recognized as an influence, but I feel the results obtained, combined with the field evidence, give indications that the absorption of 20 g. in one minute is above the optimum rate if water penetration through masonry is the criterion. I believe also the evidence indicates that the 40 g. limit may be much too high for best results in the field. The field data definitely indicate the danger of an extremely low rate of absorption. It should be noted that both the optimum rate of absorption and the limits given for best results as stated by Messrs. Palmer and Parsons[1] were obtained with masonry units where the bricks were laid with a short time interval between the laying of the mortar and the brick and with unusual pressure. It is certainly conceivable that these test practices produced results different from those obtained in the field with brick laid in the ordinary manner.

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[1] *Loc. cit.*

[2] *Loc. cit.*

It should be recognized that the data in this paper are limited in amount. The evident inconsistencies apparent with any interpretation may be explainable on the basis of the effect of abnormal variations in a very limited number of tests. It is also possible, however, that the inconsistencies are a result of one or more variable influences which have not yet been discovered or fully evaluated. It is doubtful if the influence of variations in the pressure used in laying brick and the variations in brick masonry materials have been fully comprehended.

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[1] *Loc. cit.*

I believe that the rates of penetration obtained by Messrs. Palmer and Parsons, when analyzed, do not contradict the indications of my own field data regarding the selection of materials. It is my opinion that the combination of these data with the results of other investigations indicate that to give the greatest assurance of moisture-proof brick walls the brick should have a low to moderate rate of absorption, probably somewhere between 5 and 20 g. absorbed in one minute, and the mortar should have a high water-retaining capacity and the capacity to form an adequate bond layer.

MR. CLOYD M. CHAPMAN.[1]—I should like to ask the authors whether there was any difference between the technique of laying the brick in the wallettes and the usual practice followed in the field. Ordinarily a mason deposits a ridge of mortar along the center line of two or three brick, presses the next course down on this ridge of mortar until a portion squeezes out at the sides. In laying the wallettes I understand the mortar was applied to one brick at a time. Was this mortar laid as a ridge in the middle of the brick and squeezed down by the superimposed brick, or was it spread on and flattened by the trowel before laying on the next brick? Depositing mortar in a ridge along the middle of the brick and squeezing it down with the brick being laid will result in fewer air pockets than when a part of the spreading is done with the trowel. The extent of air pockets may be quite significant.

Air pockets are more likely to occur on top of the mortar than under it, and if air pockets account for some of the loss of contact or bond area, the technique of laying and its possible effect on air pocket formation and on bond area should be carefully studied.

MR. J. H. MALLON.[2]—There is one point which I think should be brought out in connection with the paper by Messrs. Palmer and Parsons. In Mr. Palmer's paper on "The Rate of Stiffening of Mortars on a Porous Base," appearing in the September 10, 1932, issue of *Rock Products*, he showed that (1) mortars in which the cementing material is portland cement without other material have the lowest water-retaining capacity; (2) mortars in which the cementing material is quicklime (slaked for a period of one week before use) and mortars in which the cementing materials are masonry cements made from natural cements and modified by the addition of stearates at the factory have the highest water-retaining capacity; (3) mortars in which the cementing material is hydrated lime, not soaked before mixing, have almost as low water-retaining capacity as mortars made with portland cement alone; and (4) mortars in which the cementing material is hydrated lime, soaked 24 hours before mixing, have higher water-retaining capacity than mortars made with hydrated lime not soaked. Now, in the wallettes described in the present paper, all the hydrated lime used in the mortar was soaked before mixing. The mortars made with portland cement and hydrated lime mixtures would have had considerably lower water-retaining capacity if the hydrate had not been soaked before mixing.

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[1] Consulting Engineer, New York City.

[2] Louisville Cement Co., Louisville, Ky.

The data in Table III of the present paper indicate that the brick has a great deal more to do with water penetration than does the mortar. This point should not be overlooked.

Although, with very careful bricklaying, the author was able to secure a good bond with impervious brick, it would seem wise in actual construction to avoid using a highly impervious brick throughout the wall. If the wall is built entirely with brick of low absorption, any water that enters through cracks is apt to go right on through the wall. But if the brick has a certain amount of absorption it will hold back the water and later, when the sun hits the face of the wall, the moisture will evaporate out again, almost as though the wall were breathing. Everything considered, it seems wise to avoid brick of extremely low as well as brick of extremely high absorption.

MR. H. F. GONNERMAN[1] (*presented in written form*).—Messrs. Palmer and Parson's paper reports results of tests on a very controversial subject. The results presented are of such a nature as to lead to differences in interpretation, particularly as regards what portions of the results should receive the most emphasis. It is hoped that the publication of these data and the discussion which they develop will lead to a better understanding of the many variables involved and thus result in benefit to the users of masonry materials.

The results as reported in this paper are somewhat at variance with Mr. Palmer's previous paper "Water Penetration Through Brick Mortar Assemblages,"[2] published in September, 1931, both as regards the method used in obtaining quantitative results and the conclusions.

Both in the present paper and in the major series of tests reported in *Research Paper No. 683* of the National Bureau of Standards,[3] the authors have placed considerable emphasis on the mortar property "water-retaining capacity" and the "extent of bond" between the mortar and the brick. Water-retaining capacity is a newly emphasized property based on tests conducted by the authors as part of this general investigation. To it they have ascribed considerable importance, not only as regards its effect on water penetration but also as regards its effect on extent of bond between the mortar and the bricks and as a measure of mortar workability.

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[1] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

[2] L. A. Palmer, "Water Penetration Through Brick Mortar Assemblages," *Journal*, Clay Products Inst. of Am., Vol. I, No. 1, p. 19 (1931).

[3] *Loc. cit.*

Based on a study of the tests reported by the authors in the various papers they have presented on the investigation, it is believed that the importance of this property and its effect on the desirable properties of a brick assembly has been over-emphasized. The desirable properties of brick work may be briefly stated as water-tightness, strength, both in compression and flexure, and a strong, durable bond which is a necessary corollary to the attainment of the other two properties.

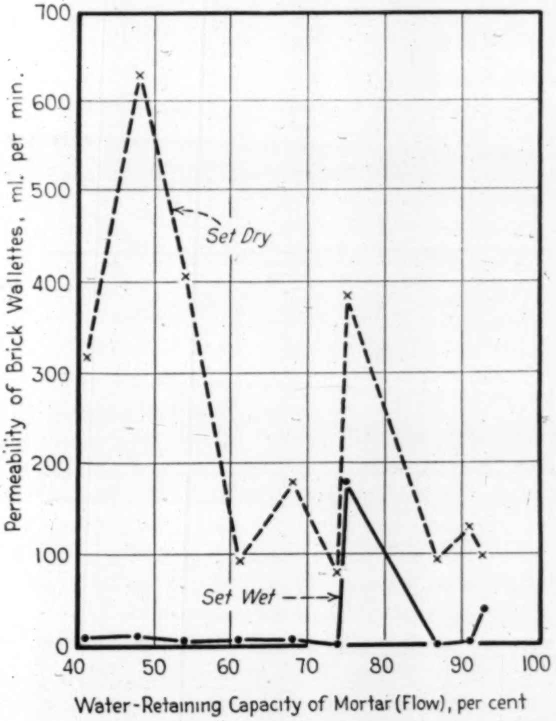

FIG. 1.—Relation Between Permeability and Water-Retaining Capacity of Mortar. Brick No. 1 set wet and dry.

Data from Table III of Palmer and Parsons paper.

The authors' conclusion regarding water-tightness states that water-retaining capacity was the most important mortar property affecting water-tightness. In the text of the paper, however, it is stated that this was not true for three of the eight conditions studied.

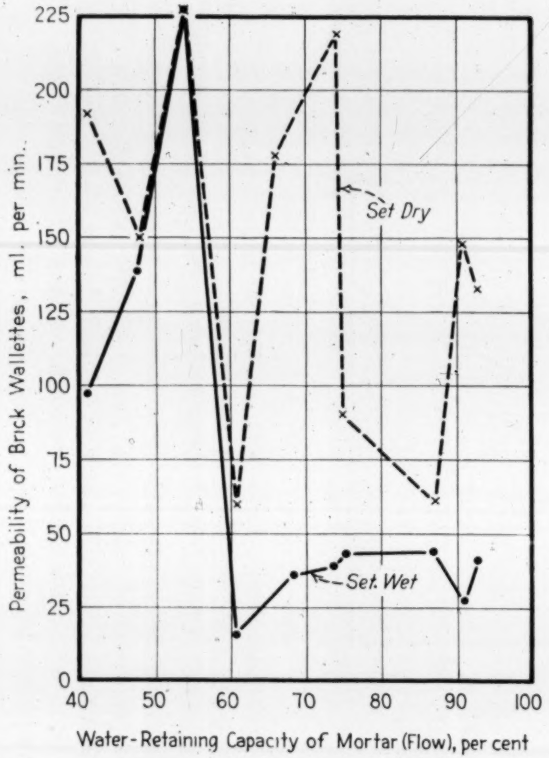

FIG. 2.—Relation Between Permeability of Brick Wallettes and Water-Retaining Capacity of Mortar. Brick No. 2 set wet and dry.

Data from Table III of Palmer and Parsons paper.

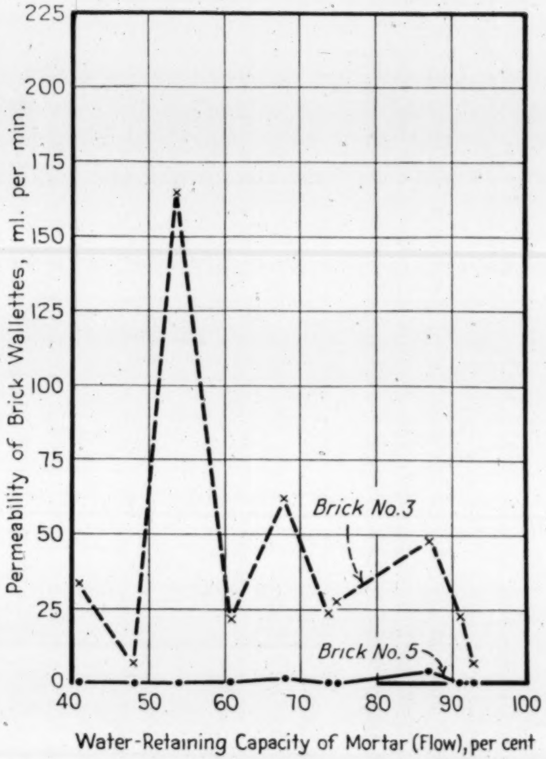

FIG. 3.—Relation of Permeability of Brick Wallettes to Water-Retaining Capacity of Mortar. Brick Nos. 3 and [illegible] set dry.

Data from Table III of Palmer and Parsons paper.

Two of the conditions studied involved the use of badly cracked brick (brick No. 6), set both wet and dry, and the excessive leakage obtained with all mortars was ascribed to the cracks in the brick. The omission of these two conditions reduces to six the number of conditions where mortar properties may be a governing factor. According to the authors, then, half of these six conditions are not in accord with their conclusions. The accompanying Figs. 1, 2 and 3 show the relationships, for these six individual conditions, between the permeability and the water-retaining capacity of the mortars used.

The plotted points in Fig. 1 show the relationship for wallettes constructed with brick No. 1 set wet and dry. The general trend of the curve for this brick set wet indicates that the permeability decreased slightly as the water-retaining capacity increased. However, the two mortars with the highest water-retaining capacity are not superior to mortars having low water-retaining capacity. The curve for brick No. 1 set dry indicates that after a water-retaining capacity of about 60 is reached, there was no decrease in permeability with increase in the water-retaining capacity of the mortar. This figure does bring out, however, the great reduction in permeability that can be obtained by the simple and common practice of wetting absorbent brick prior to placing in the wall.

Figure 2 shows the relation between the permeability of wallettes built with brick No. 2 set wet and dry. The plotted points for this brick set wet indicate that after a water-retaining capacity of about 60 is reached there is a trend toward an increased permeability with increase in water-retaining capacity of the mortar. In the case of brick No. 2 set dry the plotted points do not indicate a satisfactory correlation between water-retaining capacity and permeability to warrant definite conclusions. This figure, however, again emphasizes the desirability of wetting absorbent brick before placing in the wall in order to obtain water-tightness. Wetting produced a reduction in the permeability for all but one of the mortars used with this brick.

In Fig. 3 are plotted relationships between permeability and water-retaining capacity of mortars using bricks Nos. 3 and 5 set dry. In this case there is no correlation between the water-retaining capacity and permeability for either of the test conditions.

From the relationships brought out in Figs. 1, 2 and 3 it appears that the authors' conclusions are not generally true for five of the six conditions studied in so far as they pertain to the influence of water-retaining capacity of mortar on water transmission through brick assemblages. On the other hand, by wetting absorbent brick a marked reduction in permeability was effected.

It is of interest also to note the effect water-retaining capacity had on bond durability. The authors refer to water-retaining capacity of mortar as influencing the "extent of bond." They considered extent of bond as the bonded area. This was *estimated* from visual inspection of broken specimens. This procedure apparently ignores the several physical measures of extent of bond which were available from the data. These were:

1. Strength of bond between two-brick assemblies after 1 year's wetting and drying cycles.

TABLE I.—MORTAR PROPERTIES AND BOND DURABILITY.

Data for Column *1* Supplement to National Bureau of Standards *Research Paper No. 683*, May, 1934.
Data for Column *2* Table 10 of National Bureau of Standards *Research Paper No. 683*, May, 1934.
Data for Column *3* Table 13 of National Bureau of Standards *Research Paper No. 683*, May, 1934.
Column *4* a composite percentage of columns *1*, *2* and *3*.

Mortar	Water-Retaining Capacity (Flow)	Bond Durability of Brick and Mortar Specimens (all Bricks)				
		1	*2*	*3*	*4*	*5*
		Strength of Bond After 1 Year Wetting and Drying, lb. per sq. in.	Average Number of Specimen Cycles Before Bond Disruption, During 35 Cycles of Freezing and Thawing	Average Transverse Strength of Brick Beams at 1 Year, lb. per sq. in.	Composite of Columns 1, 2 and 3, per cent of Best Performance	Bond Durability Ranking
A I	45	63.3	105	126	82	2
XTd	54	80.8	105	161	100	1
XRa	75	53.0	79	115	71	4
XVa	84	57.5	91	102	73	3
XVb	93	41.6	47	65	45	9
XRc	91	26.0	31	42	29	10
C I	90	7.0	12	14	10	15
C II	68	17.1	34	37	25	12
C III	71	10.2	15	18	12	13
C IV	96	13.0	10	17	11	14
B I	74	26.7	85	43	47	8
B III	41	32.9	105	50	57	7
B IV	48	49.4	105	49	64	6
B VI	61	40.0	105	91	70	5
B XI	87	13.9	51	19	26	11
CONSOLIDATED AVERAGE OF MORTARS WITH 5 LOWEST AND 5 HIGHEST WATER RETAINING CAPACITIES						
A I, XTd, B III, B IV, B VI	50	53.3	105	95	75	4
B XI, C I, C IV, XVb, XRc	91	20.3	30	31	24	13

2. Durability of bond after 35 cycles of freezing and thawing extending over a 1-yr. period.

3. Flexural strength of brick beams at age of 1 year which had been subjected to a number of wetting and drying cycles.

The authors' data on these three properties, as taken from *Research Paper No. 683*, are assembled in the accompanying Table I. In order to obtain a numerical measure of bond durability, the values for strength of bond, column 1, resistance to freezing and thawing, column 2, and transverse strength of brick beams, column 3, of Table I for the individual mortars, were expressed as a percentage of the corresponding values for the mortar showing the best performance in each of the three columns. The average of these percentages for each mortar is shown in column 4. These averages are used as a numerical measure of bond durability.

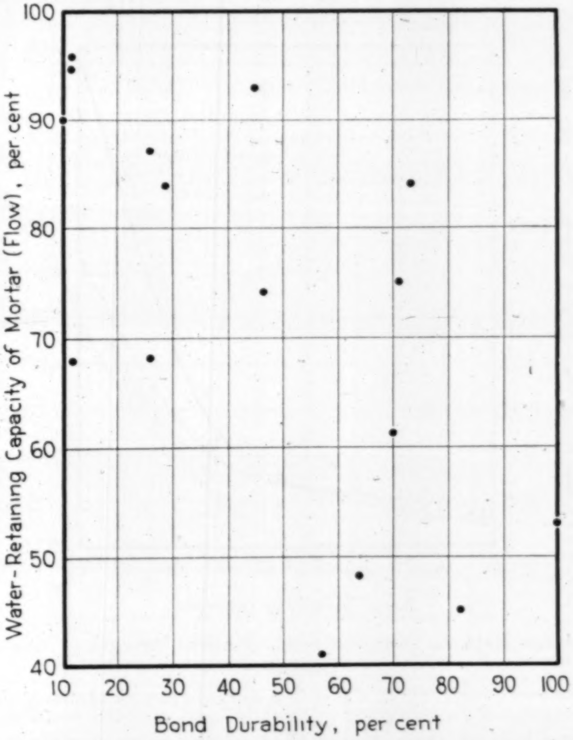

FIG. 4.—Relation Between Water-Retaining Capacity of Mortar and Bond Durability.

Data from National Bureau of Standards *Research Paper No. 683*, May, 1934.

On this basis, mortar XTd is rated as 100 per cent in strength of bond, 100 per cent in resistance to disruption of bond in 35 cycles in freezing and thawing, and 100 per cent in average transverse strength of brick beams at one year. Therefore, it had the highest bond durability rating of any of the 15 mortars studied. The ranking of the other mortars in respect to bond durability is shown in column 5. It will be seen that mortar A1 ranks second, and that XVa and XRa, the two 1:1:6 mortars, rank third and fourth respectively in bond durability. At the botton of Table I are average values for the five mortars having the lowest and the five mortars having the highest water-retaining capacities. It will be seen that in each instance the strength of bond, resistance to bond disruption in freezing and thawing and the transverse strength of brick beams made with the five mortars having the lowest water-retaining capacities is two and one-half to three times that of the corresponding values for specimens made with the five mortars having the highest water-retaining capacity.

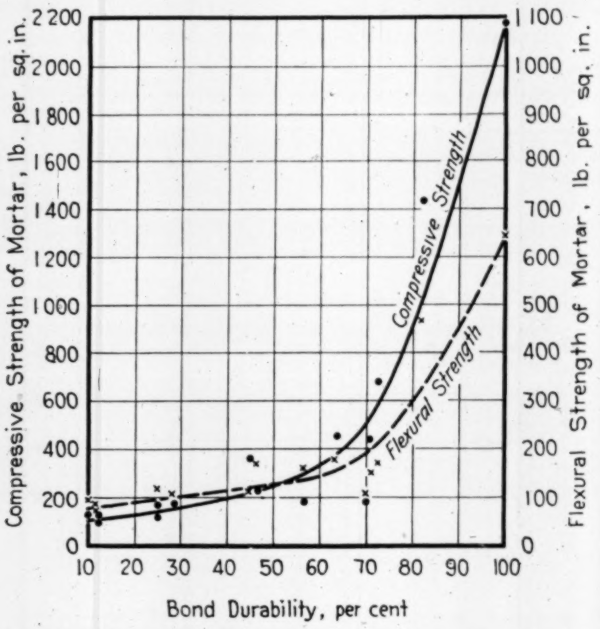

FIG. 5.—Relation Between Compressive and Flexural Strength of Mortar and Bond Durability.

Data from National Bureau of Standards *Research Paper No. 683*, May, 1934.

The accompanying Fig. 4 shows the relation between the water-retaining capacity of mortar and bond durability. There appears to be no correlation between these two properties, but the general trend of the points indicates that as the water-retaining capacity of mortar decreases the bond durability increases.

In this connection it is also of interest to note the relationship existing between bond durability and the compressive strength of the mortar as expressed in the accompanying Fig. 5. The curve drawn through the plotted points indicates quite definitely that as the compressive strength of the mortar increases the bond durability also increases. That flexural strength of the mortar had a similar effect on bond durability as compressive strength is also apparent from Fig. 5. As the flexural strength of the mortar increases, bond durability also increases.

The mortars studied which had high water-retaining capacities also generally had high shrinkage during early hardening. Such mortars

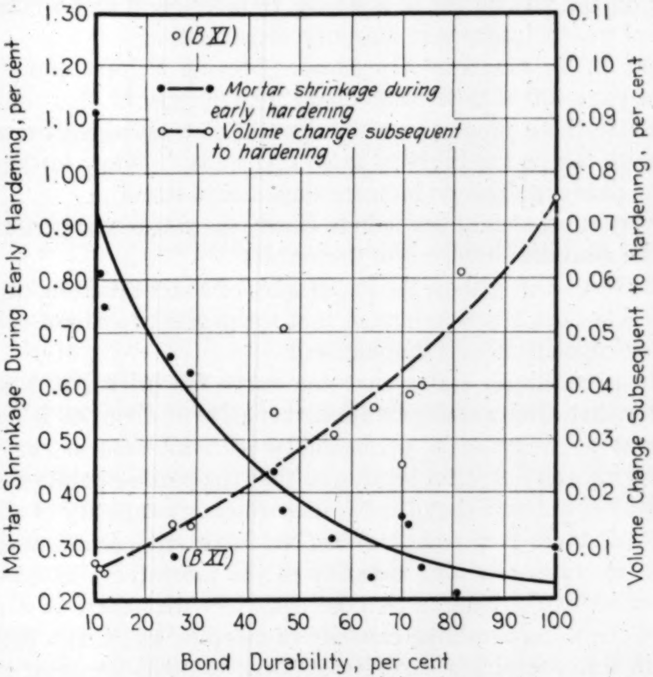

FIG. 6.—Relation Between Bond Durability and Volume Change of Mortar.
Data from National Bureau of Standards *Research Paper No. 683*, May, 1934.

when applied to brick assemblies had low bond strength, poor resistance to freezing and thawing, and medium to low transverse strength in brick beams. The accompanying Fig. 6 shows the relation between mortar shrinkage during early hardening and bond durability. It will be noted that as the shrinkage during early hardening increases, the bond durability decreases.

The effect of low volume change subsequent to hardening, another mortar property associated with high water-retaining capacity, is also shown on this figure. The curve for volume change subsequent to hardening of the mortar (the maximum value being less than 10 per cent of the maximum value during early hardening) shows that as volume change increases, the bond durability also increases. Undoubtedly this fact is due to other mortar properties associated with volume change subsequent to hardening, but for the scope of these tests high volume change subsequent to hardening cannot be said to be detrimental to bond durability.

It is believed that these figures bring out in a striking manner the relatively unimportant influence of the water-retaining capacity of a mortar on the attainment of a strong, durable bond and a maximum degree of water-tightness in masonry construction.

MR. A. T. MALMED[1] (*by letter*).—Messrs. Palmer and Parsons state on page 420 of their paper that "The purpose of this study was to correlate those properties of bricks and mortars which control the bond, with the permeability of small test walls." They further state that the data obtained do indicate such a correlation.

Every effort should be made to clarify the status and performance of all the materials involved in these tests.

The tests clearly show the importance of water-retaining capacity, but I feel as does Mr. Gonnerman that the degree or amount of water-retaining capacity is over-emphasized.

Mr. Gonnerman states that the curve for brick No. 1 set dry indicates that after a water-retaining capacity of about 60 is reached, there was no decrease in permeability with increase in the water-retaining capacity of the mortar, and that the plotted points for brick No. 2 set wet indicate that after a water-retaining capacity of about 60 is reached, there is a trend toward an increased permeability with increase in water-retaining capacity of the mortar. I assume from Mr. Gonnerman's remarks that he considers that there is a critical point in the water-retaining capacity of mortars, and that a mortar of too high water-retaining capacity acts the same as a mortar of very low water-retaining capacity. There must be some measure of truth in Mr. Gonnerman's contention because the results in Table III of the paper clearly point to this conclusion. In Table III tests are reported on 8 conditions, though only 5 brick were used. Two of the conditions studied involved the use of badly cracked brick No. 6 set both wet and dry, and the excessive leakage obtained with all mortars was ascribed to the cracks in the brick.

Now let us eliminate brick No. 6 set wet and set dry, reducing the number to 6 conditions or 6 tests. If the values added horizontally across the columns are then divided by 6 in order to secure the average for the 6 conditions the average will show the following order respectively; which table is a rearrangement of Table III of the paper with brick No. 6 eliminated:

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[1] President, Hy-Test Cement Co., Philadelphia, Pa.

Mortar	Flow After 1 min. Suction on Standard Porous Base, per cent	Brick No. 1		Brick No. 2		Brick No. 3	Brick No. 5	Average for Each Mortar, ml. per min.
		Set Wet, ml. per min.	Set Dry, ml. per min.	Set Wet, ml. per min.	Set Dry, ml. per min.	Set Dry, ml. per min.	Set Dry, ml. per min.	
B VI..........	61	7.5	94.0	15.0	60.5	21.0	0.0	33.0
B XI..........	87	0.0	95.0	44.0	60.5	48.5	4.0	42.0
XVb..........	93	40.0	98.0	40.0	132.0	6.0	0.0	52.7
XRc..........	91	5.5	128.0	26.5	149.0	23.0	0.0	55.3
B I..........	74	2.5	77.5	39.0	217.0	24.0	0.0	60.0
C II..........	68	6.0	178.0	36.0	178.0	61.0	1.0	76.7
B III..........	41	10.0	317.0	97.0	192.0	33.5	0.0	108.3
XRa..........	75	177.0	386.0	43.0	90.0	27.0	0.0	120.5
B IV..........	48	13.0	626.0	138.0	150.0	5.5	0.0	155.4
XTd..........	54	7.5	402.0	226.0	226.0	165.0	0.0	171.1
Average for each brick ..		27.0	240.0	70.5	146.0	41.5	0.5	

It will be observed that mortar B VI with a flow of 61 is at the top of the list showing an average of only 33 ml. of water transmission per minute for the 6 tests.

Mortar XRc with a flow of 91 showed 55.3 ml. of water transmission per minute for the 6 tests.

Mortar XVb with a flow of 93 showed 52.7 ml. of water transmission per minute for the 6 tests.

Most certainly there must be some other condition besides "high" water-retaining capacity that enabled mortar B VI with a flow of 61 to show up to such greater advantage than mortar XRc with a flow of 91 and mortar XVb with a flow of 93.

If there was but a slight difference in the number of milliliters between mortars B VI, XRc and XVb the results could be attributed to variable conditions in the laboratory, but it is noted that 52.7 ml. is 60 per cent more water than 33 ml., and 55.3 ml. is 67 per cent more water than 33 ml. passing through the brick wallettes. This is certainly a wide difference in the permeability between mortars B VI on the one hand with a water-retaining capacity of 61, and mortars XRc and XVb with water-retaining capacities of 91 and 93 respectively.

It therefore seems to me that although water-retaining capacity undoubtedly is an important factor, it would be inexpedient to overemphasize its importance until definite tests, similar to these, prove otherwise. We ought to eliminate opinions and let the cold figures of a testing machine tell their own story.

Messrs. L. A. Palmer[1] and D. A. Parsons[1] (*authors' closure by letter*).—The readers will note in Table III that for brick No. 6 (badly cracked) the average rates of water transmission were highest for the group of mortars of low water-retaining capacity (mortars B III, B IV and XTd). The fact that water could penetrate through the cracks in the bricks did not annul the fact that it could also pass between bricks and mortar where there was no adhesion. The presence of cracks in bricks raised all values of course, but did not alter comparisons. There is therefore no reason for excluding brick No. 6 from consideration.

In the accompanying Table II, the flows of the mortars after suction may be compared with the average maximum rates of water penetration through the test walls with brick No. 6 (averages of data of both columns 9 and 10, Table III of the paper) and the ratios, tensile bond strength with brick No. 6 set dry to tensile bond strength brick No. 6 set wet (15 minutes total immersion), computations of data given in supplement to National Bureau of Standards *Research Paper No. 683*.

Table II.—Tensile Bond Strength and Flow of Mortars After 1 min. Suction as Related to Wall Permeability. All Data Obtained with Brick No. 6.

Mortar	Flow After 1 min. Suction on Porous Base, per cent	Average Maximum Rate of Water Transmission Bricks Set Both Dry and Partially Wetted, ml. per min.	Average Tensile Bond Strength at 3 Months		Ratio, Bond Strength Brick Dry, to Bond Strength Brick Wet
			Brick Set Dry, lb. per sq. in.	Brick Set Wet, lb. per sq. in.	
XVb	93	277.5	27.9	24.0	1.2
XRc	91	166.0	21.2	19.3	1.1
B XI	87	233.5	14.2	12.7	1.1
XRa	75	250.0	34.3	32.6	1.1
B I	74	253.5	10.0	22.7	0.5
C II	68	522.5	8.4	19.0	0.4
B VI	61	247.0	27.9	30.0	0.9
XTd	54	731.0	16.1	67.3	0.2
B IV	48	867.5	8.4	20.6	0.4
B III	41	623.5	8.8	35.4	0.3

Very likely, the ratio, tensile bond strength with porous bricks laid dry to tensile bond strength with porous bricks laid wet, is a better index of water-retaining capacity of mortars than the flow after suction on a porous base. On the basis of this ratio mortar B VI had a water-retaining capacity (ratio 0.9) comparable to that of mortars XRc and B XI (ratio 1.1). A great deal may be said in criticism of the flow after suction on a porous base as a measure of water-retaining capacity and criticism of the method of measuring this property is more to the point than criticism of the importance assigned to the property. This is with reference to both Mr. Malmed's and Mr. Gonnerman's comments. The value, flow of 60 per cent after 1 minute suction, may be taken too seriously if it is not realized that the authors had to devise their own approximate measure of water-retaining capacity and that the method has by no means been perfected.

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[1] Research Associates for the Research Committee of the Mortars and Masonry Research Fellowship supported by the American Face Brick Assn., the National Lime Assn., the Portland Cement Assn., and a group of manufacturers of masonry cements.

In the accompanying Table II, with the exception of mortar B I, which was no doubt over stearated and somewhat erratic generally, the correlation between relatively high values in the last column and low values in the third are fairly good. The same applies to the data obtained with porous brick No. 1.

As to extent of bond, it is obvious that when the tensile bond strength at 3 months with portland-cement mortar, XTd, was only 16.1 lb. per sq. in. (Table II) that the distribution of adhesion was very poor. The low ratios in the last column, Table I above, were due to poor extent of bond. Such values are good physical measures of this condition.

The data presented in the paper, "Water Penetration Through Brick-Mortar Assemblages," by L. A. Palmer, published in the September, 1931, issue of the *Journal* of the Clay Products Institute, should be compared with those data obtained with brick No. 1 (set wet) in the present article. This comparison can be made wherein conditions of tests were very similar in the two studies, and the agreement is good. In all other cases, the conditions of tests presented in the present paper are very different from those described in the previous paper published in 1931 in the *Journal* of the Clay Products Institute, and comparisons between the two sets of data cannot be made.

In the present paper there is no attempt to relate water-tightness of masonry to bond durability in laboratory freezing and thawing tests, compressive strength of masonry, etc. The work might well be extended so that any relationship between such properties and wall permeability may be set forth. It is known, however, that masonry, rather weak in compression, has remained water-tight and that there have been leaky walls with bricks and mortars of very high strength. In fact Mr. Connor's data[1] indicate that with mortars of outstanding strength there tends to be a maximum of separation cracks between units and mortar.

If the rate of absorption of the bricks is not reduced by wetting, then a mortar of high water-retaining capacity is desirable, as the data of the accompanying Table II indicate. Furthermore, poorly workable mortars tend to undergo segregation when placed on a saturated or impervious unit. This segregation tends to interfere with getting good bond distribution. That it may not have affected the data presented in the present paper may have been due to the fact that the procedure followed in making the specimens was such as to limit the time during which segregation could occur. In National Bureau of Standards *Research Paper No. 683*[1] (Table 13) it may be noted that in some cases low and even zero values for strength of masonry beams in flexure were obtained with dense cement mortars of high strength. It is pointed out in that paper that this condition was due to segregation in the mortar followed by volume changes in the mortar subsequent to its hardening.

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[1] C. C. Connor, "Resultant Separation Cracking Between Various Mortars and Brick in Existing Brick Structures," see p. 454.

Mortars of low water-retaining capacity are mortars of poor workability. According to Warren E. Emley's statement in National Bureau of Standards *Technologic Paper No. 169:* "The ability to retain water, while the most important, is not the only factor governing plasticity." Being the most important factor controlling plasticity, the two properties must, however, be closely related. If water-retaining capacity is not highly necessary, then neither is workability or plasticity.

Mr. Gonnerman's Figs. 1, 2 and 3 show that the maximum rates of water transmission were obtained with mortars of low-water-retaining capacity. No more than this has been stated by the authors.

The last paragraph of Mr. Connor's discussion refers to the importance of the properties, water-retaining capacity of mortars and absorption rate of bricks and indicates that the data in the present paper and his own observations are in general agreement with respect to these properties.

It has been the opinion of some that mortar resistance to laboratory freezing and thawing is related to water-tightness of unit masonry. The present paper is, however, not concerned with assumptions. The authors obtained no data indicating such a relationship. Possibly Mr. Gonnerman assumes that water-tightness is directly dependent on bond durability as determined by laboratory freezing and thawing if his Table I has any direct bearing on the subject. In the light of present knowledge there is no good basis for such an assumption. Two factors control the resistance to frost in finished unit masonry structures. These are: the extent to which the wall becomes saturated during its life history and the frost resistance of the building materials, units and mortars. Laboratory freezing-and-thawing tests eliminate the first factor, which is actually more important than the second. In actual buildings, frost resistance of materials that are so well bonded together that the wall remains comparatively dry is, for the most part, superfluous. Frost resistance of materials in a wall that is frequently saturated again and again is useless and of small comfort to the occupant of the building. The condition, damp interiors, is more troublesome than the slow disintegration of the materials by frost. Furthermore, if the exposure of the wall that admits water freely is abnormal, disintegration is inevitable, regardless of frost resistance of materials. Frost resistance can hardly compensate for the condition, frequent and excessive saturation of the wall. In freely exposed places, parapet and retaining walls, etc., frost resistance of materials is relatively of greater importance than in the bulk of buildings generally.

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[1] *Loc. cit.*

It must be also borne in mind that in the laboratory study of bond durability, all porous bricks were wetted when laid. With bricks laid dry the results would have been far different than those recorded.

According to H. Kreuger and the British Building Research Station, buildings from two to five centuries old, constructed with mortar and stone and brick of those days have weathered well in the climates of Sweden and England. This could not have happened had the walls been excessively saturated again and again.

The flexural data plotted in Mr. Gonnerman's Fig. 5 should be compared with the data for flexural strength of brick beams in Table 13 of National Bureau of Standards *Research Paper No. 683*. We are more concerned with the flexural strength of masonry than with the flexural strength of mortar *per se*. The bond between brick and mortar is the weakest thing in most modern masonry. The strength of brick beams in flexure at three months obtained with 1 lime: 1 portland cement: 6 sand mortars was greater than that obtained with the portland-cement mortars in the case of bricks Nos. 1, 3, 5 and 6. Bricks Nos. 1 and 6 (porous) were laid wet in these beam tests. Bricks Nos. 3 and 5 were of low absorption and required no wetting. These were 4 of the total of 6 bricks studied. The transverse strength of the 1:1:6 mortar specimens was however only about one-third that of the portland-cement mortars. The reason that the mortar three times as strong did not produce a beam of corresponding strength was due to a poor bond. The mortars here referred to are among those listed in Table I of Mr. Gonnerman's discussion. It is difficult to see any possible bearing of the plot shown in Mr. Gonnerman's Fig. 5 on the subject under discussion, the bond of mortar to brick and its relation to wall permeability. In the publication by the Alton Brick Co., "Impervious Brick Masonry" (1931), on pages 67 and following it is shown that the flexural strength of brick masonry was greater with 1:1:6 than with portland-cement mortars of higher strength.

As the strength of bond is obviously related to the strength of mortar for the particular conditions chosen by Mr. Gonnerman, it is not surprising that Mr. Gonnerman's "numerical measure of bond durability," being to a major degree a function of strength of bond, gives good correlation with strength of mortar.

Those mortar properties which contribute to good bond distribution and water-tightness of masonry are usually not associated with resistance to laboratory freezing-and-thawing tests. This refers to Mr. Gonnerman's Fig. 4. However, it is again emphasized that an excessive amount of water should not enter the wall in the first place. In the laboratory freezing-and-thawing tests wherein frozen specimens were thawed by immersion in water, the fact that the masonry assemblage may have had integrity was eliminated from consideration. The same assemblage, as a part of a wall, would not ordinarily have become saturated were the bond intact, and for this reason would have composed frost-resistant masonry. In the laboratory tests, water was forced into the specimens despite any natural resistance to penetration which they may have evidenced in a wall. The procedure of immersing brick masonry in a tank of water is seldom if ever paralleled in actual construction. Such tests are fair enough to hydraulic cements, admirably adapted for the construction of dams and reservoirs, materials which develop much of their ultimate strength in a relatively short time, and manifestly unfair to many natural cements and limes which develop strength and as a consequence greater frost resistance more slowly. When it is shown that mortars in most walls are usually saturated and that the water so saturating them is alternately frozen and thawed as many as 35 times during the first year of the life of the building, then laboratory freezing-and-thawing tests will have some practical meaning. However, if that is ever shown, no one will be able to understand how there can be left standing any buildings over 10 years old (without regard to buildings centuries old, constructed with soft bricks and lime mortar and which have weathered well).

Mr. Malmed, in stressing the importance of the difference between an average of 33 ml. with B VI and 52.7 ml. with mortar XRc, quite overlooks the fact that differences even of this magnitude could easily enough be due to variations in the different individual bricks laid in the different assemblages. As previously shown, in Table II of this closure, there is no point to eliminating brick No. 6 from consideration. One should not consider that classical measurements are possible in a large number of tests in this type of work. The methods of control are not so precise as to warrant such an impression. Also the number of tests (3) with each brick and mortar combination was small.

The criticism of Mr. Connor is very much to the point and is appreciated. He apparently realized the difficulties involved in duplicating field conditions in the laboratory.

Messrs. Mallon, Gonnerman and Connor have rightfully emphasized the importance of a low rate of absorption of the bricks at the time of laying. The test results show that, irrespective of the properties of the mortar used, the leakage of the specimens was less when bricks of moderate or high suction were wetted before laying than when they were set dry.

Answering Mr. Chapman's question, the mortar was spread over the surface of the brick and not ridged up. In the case of the highly porous brick set dry, there were depressions which may be called pockets, caused by rapid loss of water to the brick. In the case of the wetter bricks and bricks Nos. 3 and 5 of low suction, there was not to the best of our knowledge any appreciable occurrence of air or water pockets. Vertical joints were filled by forcing mortar down both by the tip and edge of the pointing trowel.

It is well in this connection to call attention to the similarity of the results herein described and those described by others. On page 58 of the 1932 report of the British Building Research Board, there occurs this statement with reference to permeability tests with brick walls:

"In all sections penetration first occurred in *almost every case, at the bottom of the vertical joints.*"

This is in good agreement with the data recorded in Table V of the paper, despite possible wide differences in the technique of experimenters.

RESULTANT SEPARATION CRACKING BETWEEN VARIOUS MORTARS AND BRICK IN EXISTING BRICK STRUCTURES

By C. C. Connor[1]

Synopsis

The amount of separation cracking occurring between brick and mortar appears to be of prime importance. This type of cracking was studied on 38 existing buildings to determine to what extent it appeared on the surface of brickwork where the workmanship was good, and whether there was any relation between the amount of separation cracking and the kinds of material used. The study included 7 different mortars and 36 different makes of brick. The amount of visible separation cracking in brick panels at each building was measured and was found to vary between 2.5 per cent and 68.3 per cent, with an average of 31.1 per cent. It was indicated that the mortar mix, the rate of absorption of the brick, and the texture of the brick surface all influenced the results. There were no definite indications that the cracking was affected by the raw material used in the manufacture of the brick. The importance of proper combinations of brick and mortar was indicated, since in all instances where the cracking was below 10 per cent it was with combinations of mortars having a high lime content and brick of a low or moderate rate of absorption.

The data obtained, when combined with facts developed by other investigators, appear to indicate that there are certain properties of brick and mortar which must occur in combination to produce a durable bond. The necessary types of brick and mortar are available to form combinations that fulfill the indicated requirements and give promise of producing brickwork with a very low percentage of cracking, if the workmanship is of good quality.

Introduction

Most investigators are agreed that cracks are a major cause of water penetration through masonry.[2,3,4,5] Experience in remedial waterproofing work has indicated that separation cracks between brick and mortar which form at the exposed surface of walls, is especially detrimental. Palmer and Hall,[6] Voss,[5] Anderegg,[2] and others, have pointed out that bond is influenced by the type of materials used, and it would appear that the amount of visible separation cracks existing in brickwork would be an important measure of the effective bond between the materials. In new construction it is highly desirable that the materials selected shall produce a minimum of separation cracking, and information regarding the amount that occurs with various materials should serve as a practical guide. Varying quality of workmanship undoubtedly influences the amount of such cracking, but if it is indicated that certain combinations consistently produce a minimum of cracking, in spite of unavoidable variations in workmanship, their use would appear desirable.

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[1] New Jersey Bell Telephone Co., Newark, N. J.

[2] F. O. Anderegg, "Water-Tight Masonry," *The Architectural Record*, Vol. 70, No. 3, p. 202 (1931).

[3] L. A. Palmer, "Water Penetration Through Brick-Mortar Assemblages," *Journal*, Clay Products Inst., Vol. 1, p. 19 (1931).

[4] L. B. Lent, "Exterior Wall Construction," *General Building Contractor*, February, 1932, p. 35.

[5] W. C. Voss, "Permeability of Brick Masonry Walls—an Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

[6] L. A. Palmer and J. V. Hall, "Durability and Strength of Bond Between Brick and Mortar," National Bureau of Standards *Research Paper No. 290* (1931).

The purpose of this study was to find answers to the following questions:

1. To what extent does separation cracking occur in existing buildings, and does it vary greatly?
2. What is the influence of various mortar mixes on cracking?
3. Do various combinations of brick and mortar influence the amount of cracking?
4. Does the surface texture of bricks exert an influence?
5. Does the raw material used in the manufacture of brick affect the cracking?
6. Does the field evidence confirm the indications of laboratory investigations, or otherwise?
7. Has the combined research of the various investigators advanced the knowledge regarding brick and mortar assemblages to a point where it is possible to advance a logical hypothesis covering the properties of those materials required for durable bond?

The answers to these questions should aid greatly in the selection of materials with a view toward preventing the penetration of water through brick walls.

Definitions of Terms:

Separation Cracking.—The term "separation cracking" or "cracking" as used in this paper refers to a condition of lack of adhesion between brick and mortar at the exposed surface of brickwork, without regard for the cause.

High-Cement Mortar.—Used in this paper to designate mortars having a lime content of less than 50 per cent of the cement by volume.

High-Lime Mortar.—Used in this paper to designate mortars containing an amount of lime equal to or exceeding 50 per cent of the volume of the cement.

MATERIALS

The mortar mixes in the buildings studied contained many varying makes of lime and cement. Both, however, were always selected from standard makes of reputable manufacturers. All cement was portland except in one case where a slag cement was used.

TABLE I.—TYPES OF BRICK STUDIED AND THEIR CLASSIFICATION BY RATE OF ABSORPTION.

Brick	Raw Material	Surface Texture on Flat Sides	Number of Tests	Rate of Absorption, ml. absorbed in 2 min.						
				Very Low Average, Under 0.05	Low Average, 0.06 to 0.15	Moderate Average, 0.16 to 0.25	High Average, 0.26 to 0.5	Very High Average, Above 0.5	Maximum	Minimum
No. 1	fire clay	smooth	4	0.0	....	....	....	....	0.0	0.0
No. 2	fire clay	rough	3	0.01	....	....	....	....	0.01	0.005
No. 3	surface clay	smooth	4	0.01	....	....	....	....	0.015	0.01
No. 4	fire clay	rough	4	0.01	....	....	....	....	0.015	0.01
No. 5	fire clay	rough	4	0.02	....	....	....	....	0.03	0.02
No. 6	fire clay	rough	4	0.02	....	....	....	....	0.03	0.02
No. 7A	surface clay	smooth	2	0.02	....	....	....	....	0.02	0.02
No. 7B	surface clay	smooth	4	0.02	....	....	....	....	0.02	0.015
No. 7C	surface clay	smooth	4	0.03	....	....	....	....	0.03	0.03
No. 8	shale	rough	4	....	0.06	....	....	....	0.08	0.04
No. 9	fire clay	rough	4	....	0.06	....	....	....	0.06	0.06
No. 10	shale	rough	4	....	0.06	....	....	....	0.08	0.04
No. 11A	fire clay	rough	2	....	0.08	....	....	....	0.09	0.08
No. 11B	fire clay	rough	4	....	0.08	....	....	....	0.09	0.07
No. 12	shale	rough	4	....	0.08	....	....	....	0.20	0.01
No. 13	fire clay	rough	4	....	0.08	....	....	....	0.10	0.05
No. 14	surface clay	smooth	4	....	0.12	....	....	....	0.19	0.06
No. 15	surface clay	smooth	7	....	0.14	....	....	....	0.33	0.04
No. 16	surface clay	smooth	4	....	0.15	....	....	....	0.19	0.08
No. 17A	surface clay	smooth	4	....	....	0.19	....	....	0.28	0.08
No. 18	fire clay	rough	4	....	....	0.20	....	....	0.25	0.14
No. 19A	surface clay	smooth	4	....	....	0.22	....	....	0.35	0.08
No. 20	shale	rough	3	....	....	0.24	....	....	0.25	0.24
No. 21	shale	rough	4	....	....	0.24	....	....	0.26	0.20
No. 19B	surface clay	smooth	4	....	....	....	0.28	....	0.39	0.24
No. 17B	surface clay	smooth	4	....	....	....	0.31	....	0.53	0.09
No. 22	fire clay	rough	4	....	....	....	0.33	....	0.41	0.26
No. 23	shale	rough	4	....	....	....	0.39	....	0.60	0.22
No. 24	shale	rough	3	....	....	....	0.41	....	0.74	0.23
No. 25	surface clay	smooth	3	....	....	....	0.49	....	0.70	0.32
No. 26	shale	rough	3	....	....	....	....	0.59	0.87	0.28
No. 27	surface clay	smooth	5	....	....	....	....	0.60	0.88	0.25
No. 28	surface clay	rough	4	....	....	....	....	0.72	1.29	0.12
No. 29	surface clay	smooth	5	....	....	....	....	0.76	1.35	0.47
No. 30	surface clay	smooth	4	....	....	....	....	0.82	1.37	0.43
No. 31	shale	rough	4	....	....	....	....	1.00	1.35	0.83
No. 32	surface clay	smooth	3	....	....	....	....	1.05	1.42	0.76
No. 33	surface clay	smooth	3	....	....	....	....	1.14	1.76	0.75
No. 34	surface clay	smooth	4	....	....	....	....	1.24	2.08	0.57
No. 35	surface clay	smooth	4	....	....	....	....	1.30	1.76	0.94
No. 36	surface clay	smooth	4	....	....	....	....	1.42	1.80	0.87

In several instances the cement was tested in accordance with the standard method of test adopted by the Society.[1] The sand used was variable, but in the majority of cases it was a widely used building sand from Long Island, and in all cases was approved after inspection to determine its freedom from an excessive amount of loam or vegetable matter. With the exception of one case, it graded from fine to coarse and did not contain an excess of fine material. The water used was always fresh and potable.

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[1] Standard Methods of Sampling and Testing Portland Cement (C 77 – 32), 1933 Book of A.S.T.M. Standards, Part II, p. 6.

The bricks studied covered a wide range. Fire-clay, shale, and surface clay bricks of various methods of manufacture were represented. The surface texture varied from a smooth glaze on all sides to extremely rough on all sides. Some bricks had smooth but uneven surfaces; others rough flat sides and glazed faces and ends. The roughness varied in degree.

Classification of Brick:

The 36 bricks studied were classed as rough or smooth by the surface texture of the flat sides only. They were also classified by the raw material used in their manufacture, and divided by their rates of absorption into five groups: namely, very low, low, moderate, high, and very high. The division was arbitrarily made. The very low group contained those brick with rates of absorption which apparently tended to cause films of water to be formed on the bond plane when laid with mortar. The low group contained those brick which probably should have been laid dry. The moderate group probably could safely have been laid dry but a slight amount of wetting would have been beneficial. The high and very high groups apparently should have been thoroughly wetted before laying. The rate of absorption ranged from the very lowest among fire-clay brick to the highest among surface clay brick. No dry pressed brick, however, were included in the study. Table I shows the classification of the bricks studied.

Tests for Rate of Absorption:

The rate of absorption was tested in the following manner: The mouth of a small tubular vessel was carefully cemented to the exposed faces of brick selected to include the major variations in color. The vessel had a small opening at the top through which it was filled with water, and which also served as a vent. The vessel was kept filled to the top of the vent opening and the amount of water absorbed in two minutes was measured by reading the amount of water added from a burette graduated in 0.01 ml. The area of the brick exposed to the water was 0.63 in. in diameter, and was constant for all tests. Since brick in walls rarely if ever reach a constant dry weight, the rate-of-absorption tests were made after a period of two drying days in order that all surfaces should be as equal in dryness as possible. Repeat measurements were made at different times at certain buildings to check the accuracy of the initial measurements. Where the brick had glazed or rough-textured surfaces, this surface was chipped away to a slight depth and the test made on the surface exposed.

Mortars Studied:

Dependable records of the mortar mixes specified for all buildings studied were available. The mixing had always been subject to strict and competent inspection. There were six mortar mixes and one masonry cement. The mortar mixes were as follows:

One part cement, three parts sand, with 15 per cent of dry hydrated lime added.

One part cement, three parts sand, with 25 per cent of dry hydrated lime added.

One part cement, three parts sand, with 50 per cent of dry hydrated lime added.

One part cement, one part dry hydrated lime, and six parts sand.

One part cement, one part hydrated lime soaked to a putty, and six parts sand.

One part cement, one part quicklime putty, and six parts sand.

All parts were by volume and the percentage of lime added was a percentage of the volume of the cement.

The masonry cement, designated as masonry cement A, was a mixture of portland cement and dry hydrated lime which was mixed with more portland cement when used. The mortar mix developed by this procedure contained slightly more lime than cement by volume.

BUILDINGS STUDIED

Thirty-eight buildings were studied. Their ages varied from 3 to 15 years. All buildings except two were built for the same company, and the inspection at the time of erection was competent and thorough. The workmanship on all buildings was considerably better than average and, judging from the surface indications, was not greatly variable.

PROCEDURE

Selection of Panels:

Panels of brickwork containing 40 ft. of horizontal joints and 10 ft. of vertical joints were marked off for the measurement of separation cracks. Wherever possible, 4 panels were measured, one on each wall exposure. With the exception of two buildings, all panels were on the first story walls and were located in fairly large flat areas of brickwork without breaks or offsets, and free from obvious settlement cracks. All panels were between the levels of the floor slabs and none was at the level of beams. All the mortar joints measured had been tooled and all but six, concave tooled.

Method of Measurement of Separation Cracking:

The lengths of all cracks, openings or separations visible to the eye were measured except in those lengths of joint where the mortar

TABLE II.—SEPARATION CRACKING IN BRICK STRUCTURES.

Mortar Mix	Building	Brick	Number of Measurements	Cracking, per cent					Individual Maximum Measurement, per cent	Individual Minimum Measurement, per cent	Type of Brick Surface on Flat Sides	Brick Raw Material
				Very Low Rate of Absorption	Low Rate of Absorption	Moderate Rate of Absorption	High Rate of Absorption	Very High Rate of Absorption				
1:3 + 15% dry hydrated lime	No. 1	No. 1	3	39.3	....	....	....	....	57.7	17.2	smooth	fire clay
	No. 2	No. 2	2	24.2	....	....	....	....	29.5	19.0	rough	fire clay
	No. 3	No. 19B	4	....	....	....	26.0	....	35.8	16.3	smooth	surface clay
	No. 4	No. 36	3	....	....	....	....	35.5	53.5	14.8	smooth	surface clay
1:3 + 25% dry hydrated lime	No. 5	No. 5	3	68.3	....	....	....	....	73.7	65.3	rough	fire clay
	No. 6	No. 7A	4	65.2	....	....	....	....	80.8	41.2	smooth	surface clay
	No. 7	No. 7B	4	33.4	....	....	....	....	54.2	14.0	smooth	surface clay
	No. 8	No. 7C	5	52.1	....	....	....	....	72.2	19.0	smooth	surface clay
	No. 9	No. 12	4	....	26.2	....	....	....	37.5	13.2	rough	shale
	No. 10	No. 13	3	....	65.5[a]	....	....	....	76.8	55.8	rough	fire clay
	No. 11	No. 14A	4	....	27.9	....	....	....	38.8	21.7	smooth	surface clay
	No. 12	No. 16	4	....	49.5	....	....	....	69.7	16.0	smooth	surface clay
	No. 13	No. 17A	3	....	....	33.2	....	....	40.8	28.2	smooth	surface clay
	No. 14	No. 19A	3	....	....	32.7	....	....	38.8	28.3	smooth	surface clay
	No. 15	No. 20	3	....	....	57.9	....	....	59.3	57.0	rough	shale
	No. 16	No. 27	4	....	....	....	....	54.8	75.8	19.2	smooth	surface clay
	No. 17	No. 30	4	....	....	....	....	31.1	43.0	15.2	smooth	surface clay
	No. 18	No. 31	3	....	....	....	....	49.9[b]	69.5	32.8	rough	shale
	No. 19	No. 32	4	....	....	....	....	48.5	68.3	17.2	smooth	surface clay
1:3 + 50% dry hydrated lime	No. 20	No. 6	3	22.1	....	....	....	....	28.2	17.0	rough	fire clay
	No. 21	No. 10	3	....	5.3	....	....	....	6.7	3.8	rough	shale
	No. 22	No. 17B	3	....	....	....	18.6	....	32.8	9.3	smooth	surface clay
	No. 23	No. 24	3	....	....	....	14.3	....	17.5	12.0	rough	shale
1:1:6 using dry hydrated lime	No. 24	No. 9	4	....	12.3	....	....	....	23.2	2.8	rough	fire clay
	No. 25	No. 18	3	....	....	6.0	....	....	7.2	5.2	rough	fire clay
	No. 26	No. 21	3	....	....	5.1	....	....	11.7	0.7	rough	shale
	No. 27	No. 22	4	....	....	....	49.5	....	57.3	42.0	rough	fire clay
	No. 28	No. 28	4	....	....	....	....	26.6	50.7	15.7	rough	surface clay
1:1:6 using hydrated lime (soaked)	No. 29	No. 4	4	18.6	....	....	....	....	25.2	13.2	rough	fire clay
1:1:6 using quicklime putty	No. 30	No. 33	4	....	....	....	....	12.6[c]	24.8	7.3	smooth	surface clay
	No. 31	No. 35	4	....	....	....	....	11.3	28.8	4.0	smooth	surface clay
Masonry Cement A	No. 32	No. 3	4	28.7	....	....	....	....	42.0	13.8	smooth	surface clay
	No. 33A	No. 8	3	....	4.8	....	....	....	8.7	2.2	rough	shale
	No. 33B	No. 15	3	....	19.3	....	....	....	25.8	12.3	smooth	surface clay
	No. 34	No. 11A	4	....	4.3	....	....	....	6.7	1.0	rough	fire clay
	No. 35	No. 11B	4	....	2.5	....	....	....	6.2	0.1	rough	fire clay
	No. 36	No. 23	4	....	....	....	51.9	....	67.2	40.2	rough	shale
	No. 37B	No. 25	2	....	....	....	42.0	....	55.8	28.2	smooth	surface clay
	No. 37A	No. 26	4	....	....	....	....	12.8	22.3	4.8	rough	shale
	No. 38A	No. 29	3	....	....	....	....	37.8	52.3	28.2	smooth	surface clay
	No. 38B	No. 34	3	....	....	....	....	48.5	58.3	42.5	smooth	surface clay

[a] Fourth story measurements.
[b] Sixth story measurements.
[c] Slag cement used.

had separated from the brick on both sides, in which case only one of the two cracks was measured. All measurements were made with a tapeline. The added amount of cracks was expressed as a percentage of the total linear measurement of the mortar joints in the panel.

Accuracy:

It should be noted that the measurements of separation cracking were approximate only. In many cases, especially where the bonding surface of the brick was rough, it was a matter of judgment as to whether or not a projecting lip of mortar was a separation. Also, while every effort was made to obtain the exact added length of cracks, some error was introduced by the use of the tapeline. In measurements repeated at different intervals on the same panels, the maximum deviation obtained was 10 per cent. Where certain measurements were checked by another operator, the maximum deviation was 11 per cent. The consistency, however, of the percentages of cracking obtained in combinations of the same mortar and brick with similar rates of absorption, indicated the relative accuracy of the measurements.

There were many variables in the brickwork of the panels measured. The kinds of sand, makes of cement and lime, quality of workmanship, exposure to weather, types of brick bond, weather conditions when brick were laid, and the wetting of the brick before laying were all variables. The type of tooling was constant except in six cases. At four buildings the mortar joints in common brick walls were struck flush; at two buildings the mortar joints in face brick walls were raked and rodded. All other mortar joints were concave tooled.

PRESENTATION OF DATA

Table I gives the raw materials, the surface texture, and rates of absorption of the 36 types of brick studied. Similar brick numbers designated by letters A, B and C indicate a similar make of brick, but used in different buildings and furnished in separate consignments. Bricks Nos. 15, 25, 34 and 36 were common brick.

Table II records the separation cracking between brick and mortar as measured at the 38 buildings. The buildings are grouped according to their mortar mix, and the table shows the percentage of cracking that occurred when the various mortar mixes were used in combination with bricks of varying rates of absorption. The surface texture of the flat sides of the bricks and the raw materials used in their manufacture are also given. Similar building numbers designated by letters A and B indicate the same building, but walls of different types of brick.

TABLE III.—EFFECTS OF VARIOUS COMBINATIONS OF BRICK AND MORTAR UPON SEPARATION CRACKING.

Average Separation Cracking	Brick and Mortar Combinations		Number of Buildings	Individual Maximum Measurement, per cent	Individual Minimum Measurement, per cent
	Brick Absorption Rate	Mortar Mix			
Under 10 per cent	low	1:3 + 50% dry hydrated lime	1	6.7	3.8
	moderate	1:1:6 using dry hydrated lime	2	11.7	0.7
	low	masonry cement A	3	8.7	0.1
From 10 to 20 per cent	high	1:3 + 50% dry hydrated lime	2	32.8	9.3
	low	1:1:6 using dry hydrated lime	1	23.2	2.8
	very low	1:1:6 using hydrated lime (soaked)	1	25.2	13.2
	very high	1:1:6 using quicklime	2	28.8	4.0
	low	masonry cement A	1	25.8	12.3
	very high	masonry cement A	1	22.3	4.8
From 20 to 30 per cent	very low	1:3 + 15% dry hydrated lime	1	29.5	19.0
	high	1:3 + 15% dry hydrated lime	1	35.8	16.3
	low	1:3 + 25% dry hydrated lime	2	38.8	13.2
	very low	1:3 + 50% dry hydrated lime	1	28.2	17.0
	very high	1:1:6 using dry hydrated lime	1	50.7	15.7
	very low	masonry cement A	1	42.0	13.8
Above 30 per cent	very low	1:3 + 15% dry hydrated lime	1	57.7	17.2
	very high	1:3 + 15% dry hydrated lime	1	53.5	14.8
	very low	1:3 + 25% dry hydrated lime	4	80.8	14.0
	low	1:3 + 25% dry hydrated lime	2	76.8	16.0
	moderate	1:3 + 25% dry hydrated lime	3	59.3	28.2
	very high	1:3 + 25% dry hydrated lime	4	75.8	15.2
	high	1:1:6 using dry hydrated lime	1	57.3	42.0
	high	masonry cement A	2	67.2	28.2
	very high	masonry cement A	2	58.3	28.2

TABLE IV.—EFFECT OF SURFACE TEXTURE OF BRICK UPON SEPARATION CRACKING.

Average separation cracking horizontal joints only.

Mortar, 1:3 + 25% Dry Hydrated Lime. Brick with Very Low Rate of Absorption, per cent		Mortar, 1:3 + 25% Dry Hydrated Lime. Brick with Low Rate of Absorption, per cent		Mortar, 1:3 + 25% Dry Hydrated Lime. Brick with Moderate Rate of Absorption, per cent		Mortar, 1:3 + 25% Dry Hydrated Lime. Brick with Very High Rate of Absorption, per cent		Mortar, 1:3 + 50% Dry Hydrated Lime. Brick with High Rate of Absorption, per cent		Masonry Cement A. Brick with Low Rate of Absorption, per cent		Masonry Cement A. Brick with Very High Rate of Absorption, per cent	
Rough	Smooth	Rough	Smooth	Rough	Smooth	Rough	Smooth	Rough	Smooth	Rough	Smooth	Rough	Smooth
29.0	26.3	10.2	11.3	25.4	13.6	19.7	22.2	5.5	6.3	2.2	6.9	4.1	12.1
....	13.2	29.1	17.7	....	12.6	....	10.7	....	....	1.8	....	....	17.3
....	20.5	....	....	....	....	....	21.2	....	....	1.1	....	....	....
Average: 29.0	20.0	19.6	14.5	25.4	13.1	19.7	18.0	5.5	6.3	1.7	6.9	4.1	14.7

TABLE V.—EFFECT OF RAW MATERIAL USED IN THE MANUFACTURE OF BRICK ON SEPARATION CRACKING.

Raw Material	Number of Buildings	Average Separation Cracking, per cent	Maximum Building Average, per cent	Minimum Building Average, per cent
Fire clay	11	28.4	68.3	2.5
Shale	9	25.4	57.9	4.8
Surface clay	21	35.0	65.2	11.3

Table III gives the various combinations of brick and mortar which produced separation cracking under 10 per cent; from 10 to 20 per cent; from 20 to 30 per cent; and above 30 per cent.

Table IV gives the percentage of separation cracking at the horizontal joints only, occurring with rough and smooth-surfaced bricks. Comparisons were made where different bricks in the same group as to rate of absorption had contrasting types of surface, but had been used with the same mortar. Many of the bricks studied had rough flat sides, but glazed ends. They were classed as rough, but the cracking on the flat sides only is given to show the true comparative results obtained. The roughness of the flat sides varied greatly in degree, and the brick classified as smooth varied considerably in their unevenness.

Table V gives the averages of the separation cracking that occurred with brick grouped as to raw material, without regard to other factors.

DISCUSSION OF DATA

Variation in Amount of Separation Cracking:

The percentage of separation cracking at the buildings studied varied over a wide range. The maximum average for any one building was 68.3 per cent, and the minimum, 2.5 per cent. The average cracking for all buildings was 31.1 per cent. This average was so high that it is not surprising that moisture penetration of walls frequently occurs. If this average of cracking existed throughout the walls of a moderate-sized two-story brick building having 10,000 sq. ft. of exposed brick walls, there would be about three miles of cracks. The amount of water that could penetrate through a crack three miles long is considerable.

The low percentage of cracking found in several buildings indicates the possibility of reducing separation cracking to such a minimum that moisture-proof walls would be assured.

As the workmanship at the buildings apparently did not vary greatly, the wide range in the percentages of cracking indicates the dominating influence of other causes for separation. In further evidence of this fact, comparisons were made between the front and rear of buildings where the workmanship might have differed in quality, but no consistent indications were obtained.

Influence of the Rate of Absorption of Brick:

The rate of absorption of brick appears to have definitely influenced the results. Very low rates of absorption always produced high percentages of cracking. This was probably due to the fact that the brick did not absorb the water, which tends to segregate out of all mortars, at a rate sufficient to prevent the forming of a film of water at the plane of contact. The low and moderate rates of absorption gave much better results with high-lime mortars, but did not consistently affect the high-cement mortars. Bricks of high and very high rates of absorption generally produced high percentages of cracking with all mortars, although the percentage at four buildings where high-lime mortars were used might be considered as moderate. The indications are that good results cannot be made certain by any particular rate of absorption for brick if no regard is given to the mortar mix. Such results are obtained only when the proper combination of brick and mortar is used. Bricks of very low, high and very high rates of absorption, however, apparently tend to increase the cracking.

Influence of the Mortar Mix:

The mixes containing one part of cement, three parts of sand, plus 15 per cent of dry hydrated lime; and one part of cement, three parts of sand plus 25 per cent of dry hydrated lime, are here considered as high-cement mortars, while all of the others are considered as high-lime mortars. The high-lime mortars consistently produced less cracking than the high-cement mortars. This was true with all groups of brick, but was especially true with those groups having low and moderate rates of absorption. The minimum measurement obtained with high-cement mortars with any type of brick was 13.2 per cent, while the minimums obtained with the 1:3 plus 50 per cent of dry hydrated lime, 1:1:6 using dry hydrated lime, 1:1:6 using quicklime putty, and masonry cement A, were 3.8, 0.7, 4.0, and 0.1 per cent, respectively. It should be noted that the high-cement mortar containing 15 per cent of lime consistently produced less cracking than the mortar with 25 per cent of lime added.

Influence of Various Combinations of Brick and Mortar:

Table III shows the beneficial effect of the use of high-lime mortars with brick having low or moderate rates of absorption, for such combinations furnished all of the average cracking for individual buildings below 10 per cent. All of the average crackings from 10 to 20 per cent were also obtained from high-lime mortars, but six buildings out of the eight in this group were of brick having a very low, high, or very high rate of absorption. The other two buildings were of brick having a low rate of absorption, one a common brick that varied greatly in the rate of absorption of individual bricks. The other brick appeared to be one which would produce excellent results, but one quite high measurement raised the average to slightly over 12 per cent. All of the crackings obtained above 20 per cent were with high-cement mortars, or high-lime mortars with bricks having a very low, high, or very high rate of absorption.

The results obtained at buildings Nos. 34 and 35 where the same brick was used with masonry cement A, a high-lime mortar, are worthy of note. The brick was a fire-clay brick, rough on the flat sides, and having a low rate of absorption. The buildings were constructed by different building contractors and the usual variables of workmanship existed, although at both the workmanship appeared to have been good. The average percentage of cracking at building No. 34 was 4.3 per cent and at building No. 35 was 2.5 per cent. The maximum measurement at the first building was 6.7 per cent and at the latter was 6.2 per cent. The consistently low cracking found at these buildings indicates that the construction of moisture-proof walls is possible if the proper combinations of brick and mortar are used with good workmanship.

While the results indicate that consistently good results cannot be assured with bricks having high or very high rates of absorption, percentages of cracking obtained with the 1:1:6 mortar using quicklime putty at buildings Nos. 30 and 31 indicate that the safe range of absorption may be widened by the use of this type of lime. It might be presumed that the fairly good results obtained at these buildings were due to the very thorough wetting of the brick, but at building No. 28 where a brick of a similar rate of absorption was wetted as thoroughly as was practicable with a hose, the cracking was more than twice as great. The mortar used at building No. 28 was a similar mix, but the lime used was a dry hydrate. It is more probable that the difference in results was due to the higher water-retaining capacity of the 1:1:6 mortar using quicklime putty than to the difference in the wetting of the brick. Palmer and Parsons[1,2] have pointed out the beneficial influence of a high water-retaining capacity in mortar on bond, and showed that with 1:1:6 mortars the use of quicklime putties produces more of that property than do dry hydrates of lime. The data obtained from existing buildings tended to support their conclusions.

The increasing spread occurring between the maximum and minimum measurements of cracking where bricks of high or very high rates of absorption had been laid with high-lime mortars, indi-

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[1] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

[2] L. A. Palmer and D. A. Parsons, "The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. xxxv, No. 18, p. 18 (1932).

cates that good results would be made more certain by using brick of a low or moderate rate of absorption which could safely be laid dry. The danger, however, of too low a rate of absorption is also indicated.

Influence of the Surface Texture of Brick:

Rough-surfaced brick produced less cracking with high-lime mortars than did bricks with smooth surfaces. The contrary was true with high-cement mortars where the smooth-surfaced bricks produced the better results. These dissimilar results probably were due to the difference in workability of the two classes of mortar. It is logical to suppose that the less plastic cement mortars bridge the indentations in a rough-surfaced brick and reduce the area of contact unless unusual pressure is applied in laying the brick. The more plastic high-lime mortars probably fill the indentations in a rough surface, thereby increasing the area of contact and forming keys with the frogs which tend to increase the resistance to stress caused by the differential volume changes occurring between brick and mortar. In general the small differences indicated that the influence of surface texture was not great.

Effects of the Raw Materials Used in the Manufacture of Brick:

Table V shows that the general average of cracking obtained was lowest for shale and highest for surface-clay bricks. It is indicated, however, that factors other than the raw materials probably were responsible for these results. At building No. 33B brick No. 15, a surface-clay brick of the common variety, produced an unusual amount of cracking for a brick having a low rate of absorption used with a high-lime mortar. The rate of absorption, however, for the individual bricks tested, varied from very low to high, and this may have affected the amount of cracking. There was no certain indication that a well-burned surface-clay brick having a uniformly low or moderate rate of absorption would not give results equal to similar bricks of fire clay or shale, if used with the proper mortar.

Correlation of Results and Conclusions with Those of Other Investigators:

It would appear that data on the bond of brick and mortar at existing buildings should furnish indications concerning the value of results obtained in laboratory investigations. The major importance of field data is that they represent results obtained under normal working conditions and are the practical evidence regarding the soundness of conclusions drawn from laboratory findings. It therefore appears desirable to attempt to correlate the data obtained in this study with some of the results and conclusions arrived at by laboratory investigators.

The fact that the field data showed that high-lime mortars containing dry lime hydrate, separated from brick having a very high rate of absorption in approximately the same proportions as high-cement mortars, but produced consistently better results with brick of a low or moderate rate of absorption, is significant. It appears to give some further indications regarding the influences of the water-retaining capacity of mortar and the bond layer upon the durability of bond.

Palmer and Parsons[1,2] indicated the importance of the water-retaining capacity of mortar. The evidence is that this property produces a workability that is retained for a period of time, which in turn increases the intimacy of contact between the mortar and brick and results in a greater extent of bond. The data of Palmer and Parsons[2] showed that a 1:1:6 mortar using hydrated lime has but little more water-retaining capacity than a 1:3 mix of portland cement with 15 per cent of hydrated lime added, as they stiffen in about the same time on a porous base. Palmer and Parson[1] also indicated that the destructive effects of the changes in volume of these two mortars were not greatly different if the extent of bond was good.

If water-retaining capacity, intimacy of contact, and differential volume changes are the controlling factors in the durability of bond, the high-cement mortars and the high-lime mortars containing dry hydrated lime should have produced approximately the same percentages of cracking with all bricks of similar rates of absorption. It is apparent from the data, however, that this was not the case. With bricks having low or moderate rates of absorption, much less cracking was consistently obtained with the high-lime mortars containing dry hydrated lime. The explanation for this difference appears to be given by Voss[3] who demonstrated the existence of a bond layer between the brick and mortar. His microscopic photographs showed that bond layers formed by 1:1:6 and 1:2:9 mortars were continuous and of considerable thickness, while those formed by a 1:3 mix of portland cement plus 10 per cent of lime by weight, which approximates 25 per cent of lime by volume, were thin and not continuous. He concluded that the bond layer probably was composed of calcium, more likely the hydroxyl than the silicate. It would appear logical to presume that at the existing buildings studied, the brick of low and moderate rates of absorption permitted the mortars to retain their water for a period of time long enough for the materials making up the bond layer to accumulate at the bond plane before loss of workability and stiffening occurred. The lime content in the high-cement mortars, however, appeared to be insufficient to form adequate bond layers.

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[1] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

[2] L. A. Palmer and D. A. Parsons, ' The Rate of Stiffening of Mortars on a Porous Base," *Rock Products*, Vol. xxxv, No. 18, p. 18 (1932).

[3] W. C. Voss, "Permeability of Brick Masonry Walls—an Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

The high percentages of cracking, found with both high-lime-hydrate mortars and high-cement mortars where the brick had a high or very high rate of absorption, appear to have a logical cause in the low water-retaining capacity common to these mortars. The resultant rapid loss of water probably caused early loss of workability, less intimacy of contact, and rapid stiffening of the mortar. These conditions apparently prevented the formation of adequate bond layers inasmuch as Voss[1] has demonstrated that the high-lime mortars have sufficient lime to produce them.

The field data indicate support for the conclusions that high water-retaining capacity and the capacity to form an adequate bond layer are both important properties of mortar in producing durable bonds, and they indicate that a low degree of either property in mortar is liable to cause a high percentage of cracking if conditions are somewhat unfavorable.

Palmer and Parsons[2] indicated that from the standpoint of durability of bond, and considering the mortars they studied, the best results could generally be obtained by keeping the rate of absorption below the value, 40 g. of water, as obtained by the partial immersion of a brick (flat side down) for one minute in $\frac{1}{8}$ in. of water.

The data obtained at existing buildings support the general thesis that for the best results the rate of absorption should be limited in range, and indicates the importance of the conclusion that the rate of absorption of brick influences the durability of bond. The data showed, however, that for mortars similar to some of those studied by Palmer and Parsons,[2] there was a lower limit below which good results were not generally obtained. One mortar did not occur in combination with brick of very low rate of absorption, but the tendency was clear.

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[1] W. C. Voss, "Permeability of Brick Masonry Walls—an Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

[2] L. A. Palmer and D. A. Parsons, "Properties of Mortars and Bricks and Their Relation to Bond," *Research Paper No. 683*, National Bureau of Standards *Journal of Research*, Vol. 12, May, 1934.

COMBINATION OF PROPERTIES NECESSARY TO PRODUCE DURABLE BOND—AN HYPOTHESIS

Many of the apparent contradications in the results and conclusions of laboratory investigators resolve when the methods used in the various tests are analyzed, and the results compared with the data obtained from existing buildings. It would appear possible and desirable to submit an hypothesis, derived from the combined indications of the results obtained in the laboratory and field, covering the properties of brick and mortar which, occurring together, will apparently produce a durable bond.

The conditions necessary for the forming of such a bond appear to be as follows:

1. Intimate and relatively complete contact between the brick and mortar, at the bond plane, which is retained during the process of early hardening of the mortar.
2. The presence of materials and properties in the mortar and brick which will produce an adequate bond layer between the brick and the main body of the mortar at the plane of contact.
3. The presence of factors that resist or reduce the destructive effect of differential volume changes between the brick and mortar, so that cracking or separation will not be caused after the mortar has hardened.

To obtain these conditions for durable bond, it appears that certain properties of brick and mortar must occur in combination. Intimate contact appears to be obtained by a combination of high water-retaining capacity for the mortar and a rate of absorption for the brick low enough to allow the mortar to retain its water for a period sufficient to achieve proper contact and prevent that contact from being destroyed by differential shrinkages due to too rapid hardening. The rate of absorption, also, must be high enough to absorb the water that tends to segregate out from the mortar and accumulate as a film or in pockets at the surface of the brick.

The formation of an adequate bond layer is apparently obtained by a sufficiency of lime in the mortar and by workability which must be retained long enough for the materials making up the bond layer to accumulate in sufficient volume at the bond plane before the mortar stiffens. The mortar probably should contain sufficient active lime after hardening for the bond layer to be added to and made more continuous by the deposition of lime salts carried in solution.

It appears that the requirements for brick to produce the formation of an adequate bond layer is essentially the same as those needed for the obtaining of intimate contact between the brick and the mortar.

The properties of mortar which resist or reduce the destructive effects of differential volume changes between the brick and the mortar appear to be low absorption, low to medium coefficients of volume change due to wetting and drying and temperature variation, good tensile strength, and those properties which, by causing intimacy of contact and the formation of an adequate bond layer, produce a relatively complete initial bond.

The requirements for brick which assist in resisting the effect of volume changes apparently are a rough surface, low or moderate rate of absorption, and the absence of any scale or shell on the surface which is not properly integrated with the body of the brick.

SUMMARY AND CONCLUSIONS

This paper records the amount of cracking that occurred in panels of brickwork at 38 buildings where six different mortar mixes, one masonry cement, and 36 types of brick were used in 38 different combinations. The data do not cover combinations of many widely used mortars and types of brick, and but sparsely cover those studied. The conclusions cannot be considered as final, but the following are indicated by the data:

1. Where brickwork is laid up with reasonable care by mechanics of average ability, the factor of workmanship does not influence the amount of separation cracking so much as do the properties of the materials used.

2. The rate of absorption of brick is important. Best results were obtained where the rate of absorption was low or moderate. Very low, high, and very high rates of absorption tended to increase the amount of cracking.

3. The effect of a high lime content in mortars was to reduce the amount of separation cracking.

4. The combination of brick with a low or moderate rate of absorption and a mortar containing a high percentage of lime consistently produced the lower percentages of separation cracking.

5. There was some evidence that quicklime putty in mortar tended to produce less cracking than an equal volume of dry hydrated lime where used with bricks having the higher rates of absorption.

6. Rough surfaces on brick tended to reduce the amount of separation cracking with high-lime mortars, but tended to increase cracking with high-cement mortars.

7. The raw material from which the brick was manufactured did not appear to affect materially the amount of separation cracking that occurred.

8. The indicated relations of the field data to some laboratory findings of others have been stated.

9. The indications of the results have been combined with the conclusions of others to formulate an hypothesis covering the requirements of brick and mortar necessary to produce a durable bond.

Acknowledgments:

The author wishes to acknowledge his indebtedness to the other investigators in the field of brick and mortar assemblages, from whose findings he has so freely borrowed. Especially is he indebted to L. A. Palmer, formerly research fellow at the National Bureau of Standards, and Prof. Walter C. Voss of Massachusetts Institute of Technology, for the results of their research and for their generous counsel during the progress of this investigation. Perry M. Moore of Maintenance Engineering Co. assisted materially with suggestions and advice.

DISCUSSION

MR. W. C. VOSS[1] (*presented in written form*).—To me the most significant fact in Mr. Connor's study is the age of the buildings and their almost complete removal from predetermined laboratory procedure. Being a survey under such conditions the results are naturally much more difficult to correlate, but nevertheless, they show some very significant tendencies. I believe the Society is fortunate that such a careful survey is now available. It puts the "proof of the pudding" test on some of our laboratory experiments.

I am raising a few questions on the paper, more to caution hasty conclusions on the part of the industry than to criticize the survey. Mr. Connor states, "It would appear that the amount of visible separation cracks existing in brickwork would be an important measure of the effective bond between the materials." This may not be rigidly true. I have had many specimens which showed marked surface separation, but excellent interior bond and contact and I would question the assumption that an external visible separation crack is indicative of complete failure in bond. If this assumption is doubtful then the error introduced by direct separation crack measurement may be material. It is however true, I believe, that such surface cracking is a physical characteristic of the mortar in conjunction with the particular brick, and as such, undoubtedly influences the behavior over the entire surface of contact.

Furthermore, I am sure that bond failures, at least sufficient to allow the entrance of capillary moisture, are often present but unrecognizable to the naked eye or even with the hand magnifier. This also I have seen so many times in the laboratory and have checked by partial immersion, that this factor further influences the accuracy with which measurements of surface cracking may be conducted, particularly if they are to indicate failure in bond over an entire brick surface.

I wish to call attention to one very important factor in Mr. Connor's paper when compared with the paper presented at this convention by Messrs. Palmer and Parsons on "Permeability Tests of 8-in. Brick Wallettes."[2] The buildings which Mr. Connor's survey includes were all more than three months old; furthermore, they were exposed to extremes of heat and cold as well as to the usual rains for several reversals of each range, at least. It would be a fallacy to suppose that, had Mr. Connor surveyed his buildings at the end of a 3-month period, even under job conditions, he would have found the same set of data.

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[1] Professor of Building Construction, Massachusetts Institute of Technology, Cambridge, Mass.

[2] L. A. Palmer and D. A. Parsons, "Permeability Tests of 8-in. Brick Wallettes," see p. 419.

The heart of this problem lies much deeper than any of the work anyone has yet done; or is perhaps at present capable of doing. What happens when a liquid comes into contact with a solid is an open field of investigation. It has been established to quite a degree in other fields that tremendous strains are set up by the phenomenon of "adsorption" and who knows whether such physical pressures and stresses do not create entirely new physical and chemical reactions.

I maintain, and there is plenty of evidence for the contention, that a bond of some sort is produced when almost any mortar comes into contact with almost any brick. Practically all tensile-bond tests show some strength in the most widely variant combinations. The question therefore is, how permanent is or can the bond be, and how continuous is it? We have only tentatively established some of the criteria for permanence and continuity of bond by various test procedures. We are now faced with the problem of "What causes this permanent and continuous bond?"

The important factors which probably affect permanence and continuity of bond are:

1. Absorption of water by brick.
2. Water-retaining capacity of mortar to control rate of movement of solubles or other particles.
3. Intimacy of immediate contact.
4. Constituents of mortar which may be expected to form a firm contact by adsorption or other methods. It must remain "Live." Generous amounts of lime do accomplish this at present.
5. Minimum differential-volume changes at least within a safe range depending upon this "bond layer" strength.

All of these are generally indicated by Mr. Connor's data and discussion. As to the amounts or composition there still seems to be considerable difference of opinion. All still point in the direction of the "Hypothesis" which the writer submitted in 1933,[1] and that higher lime contents are distinctly desirable.

MR. F. O. ANDEREGG.[2]—When any perceptible opening or lack of contact occurs between the mortar and the brick Mr. Connor is correct in viewing it with suspicion. These openings, however, seem to have resulted more from initial lack of contact, the most important cause of leakage, than from an actual opening up after hardening. If the latter were true, a crack should produce a continuous break for some distance, due to the rigidity of the mortar and of the units. Many of Mr. Connor's cracks are very short. Hence, it is suggested that the words "lack of contact" instead of "resultant cracking" would give a fairer placing of emphasis. It would be of interest to know whether the quicklimes used were high-calcium or dolomitic.

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[1] W. C. Voss, "Permeability of Brick Masonry Walls—an Hypothesis," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 670 (1933).

[2] Consulting Specialist on Building Materials, Long Island City, N. Y.

MR. L. A. PALMER[1] (*presented in written form*).—The agreement of laboratory results and observations in the field reported by Mr. Connor is in some respects very good, in others poor. On the whole, it is better than one would expect.

The existence of a bond layer at the plane where brick and mortar meet is no longer purely hypothetical. The early theory of its formation, as advanced by Mr. Voss in 1933[2] perhaps requires some modification. There is however no reasonable doubt as to its existence.

The bond layer is affected to a great extent by the speed of bricklaying and by the pressure exerted on the brick at the time it is placed on the fresh mortar and any more-or-less standardized laboratory procedure is of necessity widely different from procedures obtaining in actual construction. As a consequence there is most apt to be considerable variance between conclusions reached in the laboratory (which show what *can* be done) and results obtained in the field which indicate what normally *is* accomplished with various combinations of materials.

MR. J. H. MALLON.[3]—I should like to ask Mr. Connor whether the cracks in the buildings he inspected were evident as soon as the buildings were completed, or whether these cracks did not appear until after the walls had been subjected to seasons of alternate wetting and drying and freezing and thawing.

It has been our observation that if walls leak, they generally leak as soon as the first rainstorm hits them after they are erected. Leaks of this sort cannot be attributed to cracks formed by volume changes in the mortar subsequent to hardening.

MR. J. W. MCBURNEY.[4]—I have some questions on Mr. Connor's paper: First, has Mr. Connor any information as to whether these bricks were wet or dry at the time of the construction of the building?

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[1] Research Associate for the Research Committee of the Mortars and Masonry Research Fellowship supported by the American Face Brick Assn., the National Lime Assn., the Portland Cement Assn., and a group of manufacturers of masonry cements.

[2] *Loc. cit.*

[3] Louisville Cement Co., Louisville, Ky.

[4] Research Associate of the Asphalt and Mastic Tile Assn. at the National Bureau of Standards, Washington, D. C.

Second, will Mr. Connor define or describe "good workmanship?" Third, since many bricks have quite different rates of absorption for their flats in comparison with their faces and, since the exposed surface of a brick will pick up fairly quickly tarry matter from the atmosphere in a typical American city, I am wondering whether it is not possible that the absorption of the brick at the time of laying might not have been considerably higher than was estimated later by investigating the exposed face. Fourth, was the opinion on texture derived from the examination of the exposed face of the brick or from previous knowledge of the condition of the flat of the brick.

MR. H. F. GONNERMAN[1] (*presented in written form*).—In defining types of portland-cement-lime mortars, Mr. Connor has called 1:3 portland-cement mortars with 15 and 25 per cent by volume of lime added per volume of portland-cement, high-cement-content mortars. Similarly, a 1:3 portland-cement mortar with 50 per cent lime by volume and a 1:1:6 cement-lime-sand mortar by volume, he has called high-lime mortars. Using this terminology, the actual mortar mix referred to may be misinterpreted by the reader. This will be especially true if the conclusions are considered by themselves.

Actually the 1:1:6 mortar contains equal parts of portland cement and lime by volume. Further, 70 per cent of the cementing material by weight in this mix is portland cement. Due to this fact it is believed that it would be desirable if Mr. Connor were to substitute the actual mortar mix referred to wherever high-lime or high-cement is used in the text and conclusions.

Attention should also be called to the fact that the 1:3 portland-cement mortar mix by volume plus one-half volume of lime showed, on the average, the least separation cracking of any of the mortar types studied. This is especially interesting in view of the fact that this is the richest mix under observation and is equivalent to a 1:2 mix by volume of cementing material to sand. Apparently this has been overlooked by Mr. Connor in his discussion and conclusion.

Has any attempt been made in this study to correlate the amount of separation cracking which occurred with the age of the building or with actual water penetration into the interior of the various buildings studied?

MR. J. H. HANSEN.[2]—I should like to ask whether there has been any attempt to correlate the findings during the salvaging of second-hand brick with the findings of these investigations and experiments. I have watched laborers knocking mortar off brick, and many of them have told me that when they come to a mortar, which would be defined as a high-cement mortar in Mr. Connor's paper, they find it very difficult to get the mortar off, whereas in high-lime mortar they just knock the brick together and it drops off. This seems to indicate that the high-cement mortars have a more durable bond.

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[1] Manager, Research Laboratory, Portland Cement Assn., Chicago, Ill.

[2] Executive Secretary, Brick Manufacturers Assn. of New York, New York City.

MR. C. C. CONNOR[1] (*author's closure*).—Mr. Voss questions whether an external visible separation crack is indicative of complete failure in bond. I can state positively from experience in tearing down brickwork that it is not. There are no data on this subject but my observations have been that even where there is complete separation at the face there generally is a bond at the center portion of the brick. It not infrequently occurs, however, that in such conditions the separation continues completely around the perimeter of the flat side of the brick and forms open passages at both ends of a stretcher brick or both sides of a header brick for water to penetrate through and get into the wall. The full length of the surface crack then becomes effective as far as water penetration is concerned. The amount of separation cracking visible, certainly is evidence regarding the tendency of various combinations of brick and mortar to separate and reduce the extent of bond. This is a large part of durability of bond. Visible separation cracking also allows water to enter a wall and indicates that penetration of the wall is a possibility. Experience indicates that with high percentages of cracking this possibility is large.

As Mr. Voss states, the question of what causes this permanent and continuous bond, has not been solved but it is felt that enough is known regarding the varying influence of the properties of brick and mortar to form combinations that will produce such a bond. Materials can be selected that will give assurance of a good percentage of moisture-proof brick walls if the workmanship is similar to that now obtaining in construction of a good grade. This should not be construed to mean that further research is unnecessary. It would help greatly if the safe limits of rate of absorption of brick for best results in the field with various mortars could be more accurately established. My best judgment is from 5 to 20 g. of water absorbed in one minute, after the method of test used by Messrs. Palmer and Parsons. That range is very narrow. Further knowledge concerning bond that could be used to widen that range or make it possible to secure good results with mortars having high strength or other superior qualities would be tremendously valuable to the building industry.

Mr. Anderegg suggests that the cracking studied was due to lack of initial contact more than to opening up after hardening. Undoubtedly a certain percentage of the cracking was due to lack of initial contact but there is reason to believe that the major part was caused by volume changes after hardening. The short cracks referred to frequently showed as separate cracks in the same horizontal joint and even these were probably caused, in most part, by volume changes subsequent to hardening. Experience in remedial pointing work has clearly indicated that if two places in a horizontal mortar joint are locked fast by installing new mortar the uncracked section between may separate from the brick after a time. This is in all probability due to expansion longitudinally acting on areas of imperfect bond. It would appear likely that the short cracks were sections of imperfect bond between sections of relatively complete bond and that volume changes after hardening cracked them loose. Although there are very few measurements available, experiences at many buildings indicate that cracking increases for several years after erection. The increase in the first year or two appears to be very large proportionately but tapers off as time passes and apparently comes to an almost complete stop when the building is six or seven years old.

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[1] New Jersey Bell Telephone Co., Newark, N. J.

For example, at building No. 12, cracking was estimated to be 5 per cent 6 months after erection and 20 per cent 14 months later. All cracks in the upper story except the very narrow ones were then pointed. About two years later the cracking in the upper story was estimated at 50 per cent and many of the openings were very large. None of the pointing work had cracked. At another building careful measurements showed that too early tooling had caused cracking of 30 per cent in a certain area examined immediately after the bricks were laid. Five months later that cracking had increased to 61.7 per cent. It should be noted that at the time of laying this brickwork showed very little cracking in areas where the mortar had started to set before tooling. There is considerable evidence that most cracking occurs subsequent to the hardening of the mortar.

The quicklime used at the two buildings studied was a high-calcium lime.

Mr. Palmer's statement that laboratory research shows what *can* be done, and field results indicate what ordinarily *is* accomplished, requires a comment. In selecting materials and practices for best results in the field one consideration must always be borne in mind: that is the necessity that such materials and practices make no demands beyond the scope of workmen of moderate skill. If unusual care or workmanship is required, consistently good results cannot be expected.

Mr. Mallon's question regarding the time when the cracks appeared has been answered above. My experience has been that some walls leak very shortly after erection, more after a few months, and many within the first two or three years. Some walls have shown the first signs of water penetration as many as five years after completion.

Mr. McBurney asked whether the bricks were wet or dry when laid. That is not definitely known although it has been the general practice to hose-spray bricks of high rates of absorption, except in freezing weather, and not to wet bricks having low or very low rates of absorption.

I will not attempt to define good workmanship. It covers too wide a field. It would appear that if joints are properly filled and tooled the other details of workmanship are far less important than the properties of the materials used. Some materials apparently require less careful workmanship than others to produce good results.

The method of measuring the rate of absorption of brick has been challenged. There undoubtedly was a possibility of some small errors in the measurements because the front faces only could be tested. One source of difference between the rates of absorption of the edge and side was removed by chipping off glazed or rough faces. In general, among the types of brick tested, the wide differences between the edge and flat sides occur in those having high or very high rates of absorption. As classified, those rates of absorption cover a wide range and while it is possible that the variation between the front face and the flat side in some cases may have edged the bricks over into another classification such cases were probably rare and not particularly important in the analysis of results. The possible effect of tarry material on the face tested was eliminated where the rough or glazed surface was removed. Check measurements were always made on smooth surfaced brick in old buildings by testing where the surface had been chipped off. In only one case did the check measurement show any appreciable difference.

In determining the texture of the brick the flat side only was considered. Where necessary, a mortar joint was chipped out to expose the flat side, but generally a projecting brick could be found.

Mr. Gonnerman questioned the desirability of using the terms "high-cement mortar" and "high-lime mortar" as defined. In late years 1:3 portland cement mortars with 15 per cent or 25 per cent of lime added have been very widely used. Among builders, mortars containing appreciably more lime than those percentages are generally spoken of as high-lime mortars. The mortar mixes studied were therefore classified according to that conception and for the purposes of this paper a high-lime mortar was arbitrarily defined as one which contained an amount of lime equal to or exceeding 50 per cent of the volume of the cement.

Mr. Gonnerman points out that if the average values of cracking for each different mortar were considered, the average would be lowest for the 1:3 portland-cement mortar mix by volume plus one-half volume of lime. A study of the data shows cleared that a comparison of general averages, derived from the percentages of cracking that occured with each mortar mix and all classifications of brick, would be highly misleading and might obscure important points. The most important indication of the data is concerning the combinations of brick and mortar which produced the lower percentages of cracking. Comparison of results obtained with bricks having similar rates of absorption do not indicate that such a rich mix is necessary or particularly advantageous.

In regard to the correlation of the amount of separation cracking with the age of the buildings, no data have been given because an examination showed there was no such correlation evident. It is felt that cracking continues to increase for the first few years of a building's life if there are areas where the bond is imperfect, but apparently the amount of that cracking is a function of the properties of the brick and mortar used rather than of age. For example, Building No. 21, with the very low cracking of 5.3 per cent, is nine years old, and No. 33A, with 4.8 per cent cracking, is five years old. Buildings Nos. 12, 16, 19, 27 and 36 are all less in age but have cracking of over 40 per cent.

The correlation of cracking with actual water penetration into buildings has been asked for. That is an extremely difficult thing to do. It should be understood that there are more than 40 causes for the penetration of walls besides the cracks between brick and mortar. The practices commonly used in flashing and in the application of copings are very poor as a whole, and make parapet walls as important a source of water penetration as the cracks in brickwork. Furring, damp-proofing and thickness of walls enter into the problem and make an accurate correlation of water penetration and cracking almost impossible. Where there were large percentages of cracking and no furring on the walls, leaks generally occurred, but buildings having less than 10 per cent of cracking seldom leaked. In particular, at Building No. 21 with 5.3 per cent of cracking there was no penetration; at Building No. 25 with 6.0 per cent of cracking, no penetration; at Building 26 with 5.1 per cent, slight penetration on one wall; at Building No. 33 with 4.8 per cent, no penetration on the face brick walls, but on the common brick walls with 19.3 per cent of cracking, there was considerable penetration; at Building No. 34 with 4.3 per cent of cracking, no penetration; at Building No. 35 with 2.5 per cent, considerable penetration just below the parapet wall, apparently because the copper flashing did not cover the wall properly, causing a crack to develop completely around the building just under the coping. There were no indications of penetration on the lower floor and it is probable that there is no direct penetration through the brickwork. Buildings Nos. 5, 6, 7, 8, 9, 10, 11, 12 and several others with high percentages of cracking have leaked.

Replying to Mr. Hansen, with high-cement mortars it is true that the salvaging of bricks is frequently difficult, even where the percentage of cracking is high. The tendency of the central core or other partial areas to remain bonded and the high strength of this bond make salvage operations difficult, but this is no indication of the water-tightness of the brickwork. Some cases have been observed where brick and high-cement mortar have had almost no bond. In one case where a straight lime mortar had been used, the bricks could be pried loose with a pick and most of them were salvaged, but no cracks were found in an examination of about 150 lineal feet of joints.

THE EFFECT OF DE-AIRING DRY-PRESS AND STIFF-MUD BODIES FOR CLAY PRODUCTS MANUFACTURE

BY W. C. RUECKEL[1] AND R. B. KEPLINGER[2]

SYNOPSIS

Results of laboratory findings indicate that de-airing in connection with the dry-press process can effect many improvements. This is particularly true in regard to obtaining uniformity of properties in dry-press brick. With the present method of processing and low-pressure forming of dry-press clay, considerable variation is obtained. Laboratory investigations have disclosed that the forming pressure limitations imposed by the occurrence of cracking can be overcome by de-airing, and that brick can be formed at high pressures with improved and uniform properties.

The results of a laboratory investigation of stiff-mud bodies indicate that de-airing produced a denser and more homogeneous product. The general appearance of the evacuated ware is that the surface texture is smoother and the edges more sharply defined. Visible lamination is greatly reduced and blistering, long a problem in the sewer pipe and roofing tile industries, is entirely eliminated by the use of evacuation.

Homogeneity and increased density obtained by evacuation are reflected in a marked improvement in transverse and crushing strength, and by a lowering of the percentage absorption of each of the bodies included in the investigation.

Results obtained in commercial operation of de-airing units indicate considerable improvement in the properties of paving brick, structural tile and other heavy clay products. The effect of de-airing as reflected in the ability of structural clay products to meet A.S.T.M. specifications is also discussed.

INTRODUCTION

Clays by their very nature adsorb air very strongly. When they are blunged in water, as occurs in the slip process for the production of dinner ware and other fine whiteware products, much of this air is removed.

Clay products manufactured by the stiff-mud process or dry-press process, however, are greatly benefited by the removal of air during the forming operation. Since pockets or blebs of air produce a discontinuous mass, it is evident that de-airing should increase the homogeneity of the finished ware and result in an improvement in physical properties and in uniformity of clay products.

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[1] Research Engineer, Engineering Experiment Station, Ohio State University, Columbus, Ohio.

[2] Vice-President, Metropolitan Paving Brick Co., Canton, Ohio.

Stiff-Mud Process.—In the stiff-mud process the pulverized clays or shales are mixed with water (15 to 30 per cent) and pugged or kneaded into a consistency somewhat like that of putty. The well-kneaded particles are consolidated under pressure by the action of an auger and extruded in the desired shape from a die. In this process it is evident that much of the air is entrapped between the kneaded particles of clay and sealed in the product by the action of the auger. The air thus entrapped does not permit a perfect knitting of the clay particles and causes laminations in the finished ware.

Dry-Press Process.—In the dry-press process, the pulverized body containing 6 to 9 per cent of water is charged into the mold box of a mechanical press and formed into ware by the application of pressure. The forming pressures employed in commercial practice will vary from 500 to 1500 lb. per sq. in. Since the unconsolidated charge of clay contains 50 to 60 per cent of air, it is quite evident that considerable air may be entrapped in the formed product. The ware as it comes from the press, if sufficient air is entrapped in it, is unable to withstand the expansive action of the air and consequently pressure cracks are formed.

Considerable work on the coalescence of clay particles in the forming of ware has been done at the Ohio State University Engineering Experiment Station during the past five years. The clay products industry has found this work to be of considerable importance and is enthusiastically adopting de-airing in commercial operations.

DRY-PRESS DE-AIRING

In the Station's study of the dry-press process of forming refractories, it was necessary not only to study the properties of de-aired clay but also to work out the principles of de-airing and to develop equipment for the application of these principles.

Air in clay can be removed in three ways, namely, by subjecting the clay to repeated pressures; by evacuating the mold box; by saturating the loose clay with a gas which will be either condensed under the forming pressure, adsorbed by the clay grains, or dissolved chemically by the water film which surrounds the clay grains.

R. E. Birch[1] in an investigation conducted at the Station found that the limits of pressure employed in commercial dry-press operation varied from 500 to 1500 lb. per sq. in. In practically every instance this limit was imposed by the development of pressure cracking in the ware. The relation of forming pressure to bulk density for dry-press brick is shown in Fig. 1. The strength and porosity of the brick also vary directly with density. The distance *AE* along the abscissa represents the range of forming pressure used in commercial operation at present. It is evident that the rate of change of density along the curve *BC* is very rapid. Birch also points out that the method employed in preparing the clay batches and in charging the mold box of the press may cause the forming pressure received by the brick to vary as much as 50 per cent from its average value. From this it is evident that it would be desirable to operate at pressures greater than the point *F* where the rate of change of density with the variable forming pressure would be small. Since the curve is quite flat in the high-pressure area, brick formed under a variable pressure in the high-pressure range will have quite uniform physical properties. Uniform physical properties will mean uniform performance in refractory installations.

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[1] R. E. Birch, "Researches in Dry-Press Refractories, Part I," *Bulletin 56*, Engineering Experiment Station, Ohio State University (1930).

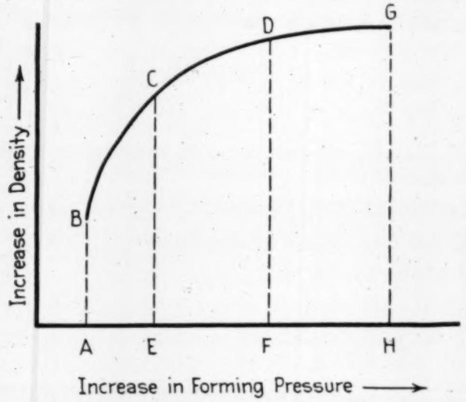

FIG. 1.—The Relation of Forming Pressure to Density.

Any attempt to control accurately the forming process by controlling the forming pressure with existing equipment would entail a great deal of difficulty. However, it is believed that a very excellent product can be made by operating the press within a range of higher forming pressure, if pressure cracking can be eliminated.

As mentioned above, three methods for eliminating pressure cracks in the forming of dry-press brick have been studied: the first de-airing by evacuation of the mold box; the second displacing the air with gas, and the third double pressing. In double pressing, the initial pressure is sufficient to force the air into pockets, the pressure is then released allowing the air to escape, after which it is repressed under sufficient pressure to heal the cracks.

Exhaustive laboratory tests were made of each of the three methods mentioned. The results of some of these tests are shown in Table I. The results of the laboratory test showed sufficient merit to warrant the application of these three de-airing methods to commercial practice. To date commercial tests have been made only on the gas de-airing and double-pressing methods. The results of these tests are shown in Table II.

TABLE I.—EFFECT OF DE-AIRING DRY-PRESS CLAY BODIES.

	Direct Evacuation				Gas De-airing		Double Pressing	
	Plastic Clay Body		Flint Clay Body		Plastic Clay Body		Plastic Clay Body	
							Single press	Double press
Forming pressure, lb. per sq. in	1500	5500	1500	5500	3500	5 500	3500	5 500
Vacuum, cm	none	50	none	50	....	....	....	....
Gas used	....	....	....	....	none[a]	Butane[b]	....	....
Fired modulus of rupture, lb. per sq. in	706	791	275	617	1642	1 849	1642	1 738
Crushing strength, lb. per sq. in	3580	3940	1395	4170	8554	10 886	8554	13 256

[a] Brick were badly laminated.
[b] Brick were free from laminations.

TABLE II.—EFFECT OF DE-AIRING ON FIRED PROPERTIES OF DRY-PRESS CLAY BATCHES.

	Gas De-airing		Double Pressing	
			Single press	Double press
Gas used	none[a]	Butane[b]	....	....
Porosity, per cent	26.63	22.88	17.53	12.90
Modulus of rupture, lb. per sq. in	359	542	848	2030
Crushing strength, lb. per sq. in	1393	1586	4023	6665
Deformation under load at elevated temperatures	1.00	0.6	....	....
Bulk specific gravity	1.90	2.00	....	....

[a] Formed under normal plant practice (low pressure forming).
[b] Formed under high pressure using butane.

It should be noted that the procedure followed in the laboratory tests was to form brick without the use of de-airing at low pressures to prevent cracking. Brick were also formed using the various de-airing methods at high pressures and this ware was free from cracking. Unde-aired ware formed at high pressures always showed some pressure cracking.

The improvement in flexural and crushing strength obtained by de-airing ranges from 10 to 50 per cent depending upon the type of body being de-aired. The increase in density and decreased porosity is also quite appreciable. A comparison of brick formed at high pressures with those formed under normal plant practice in the commercial tests indicate that the former were much denser, stronger, and more resistant to load deformation, and should be suited for refractory service where high strength and resistance to slag penetration is desired.

STIFF-MUD DE-AIRING

All the tests on stiff-mud de-airing were made in cooperation with the Bonnot Co., Canton, Ohio. The machine used in the laboratory tests was designed and constructed by the Bonnot Co. primarily for development work in designing larger machines to be used commercially. It consists of a horizontal pug mill and auger machine mounted at right angles to each other in the same plane, so that the clay discharges from the pug mill into the rear of the auger through a perforated die. Evacuation is accomplished through an opening above the junction of the two machines.

The investigation substantiated statements previously made in the literature on de-airing in that it is impossible to remove air from a plastic mass of clay until it has been broken down into relatively small pieces. Improper disintegration or shredding has been the source of erratic results obtained in all previous investigations along this line.

It is recognized that forcing plastic clay through a perforated die consumes considerable power and is probably not feasible in commercial operation. Shortly after the laboratory tests were started, new disintegrating equipment for preparing the clay in proper form was developed by the cooperator. It consumes little power and has been incorporated in the new commercial size machines. However, all bodies in the laboratory tests were processed with the original equipment.

Laboratory Investigation:

Ten different clays and shales were included in the laboratory investigation. The products manufactured from these various materials include the following: paving brick, common and face brick, roofing tile, drain tile, salt glazed brick, building tile, sewer pipe and stoneware. The bodies as secured from the plants were formed into test specimens and observations were made in order to present an analysis of the changes due to de-airing in working behavior, body structure, drying and firing behavior, as well as any changes in plastic, dry and fired strength.

The working properties of every body studied were improved by evacuation. In producing a more plastic and workable body, the effect of de-airing process is very similar to that of prolonged aging in order to induce plasticity in the mass. Dies that cannot be used in ordinary operation with certain clays can be used successfully when these same clays are de-aired. This improvement suggests the possibility of many new products. Cutting wires do not deform the evacuated column as much as they do the normal column and the ware thus has sharper edges. Blistering of the ware, long a problem in roofing tile and sewer pipe, was entirely eliminated by evacuation. The plastic and dry strength of the de-aired bodies were also considerably higher than the unde-aired bodies. Dry bulk specific gravity was increased in all cases by evacuation.

Although evacuation had no noticeable effect on color of the fired ware, considerable improvement in body structure of the fired product was noticeable (see Fig. 2). Not all of the causes of lamination are removed but healing together of the fractures is facilitated. The effect of de-airing on fired properties is shown in Table III. The data indicate that considerable improvement in the physical properties of stiff-mud bodies is made possible by de-airing. The effect of evacuation was to lower the absorption and to increase specific gravity, transverse and crushing strength and modulus of elasticity. All of the improvements are of such an order that they suggest the possibility that de-aired products will pass the requirements of the Society's specifications much more readily than will unde-aired products. The increase in bulk density ranging from 1.31 per cent to 4.58 per cent and averaging 3.2 per cent is more than offset by the increased strength of the de-aired products.

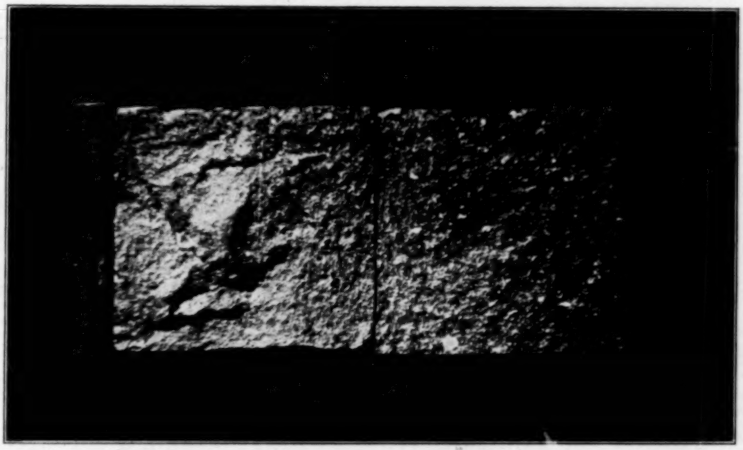

FIG. 2.—Body Structure of Unde-Aired and De-Aired Specimens of Shale After Firing.

TABLE III.—PROPERTIES OF FIRED BODIES.

Body[a]	Product	Firing Temperature, deg. Fahr.	Absorption, per cent	Bulk Specific Gravity	Modulus of Rupture, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.	Crushing Strength, lb. per sq. in.
A	Face brick	2057	2.45	2.39	4894	5 730 000	14 520[b]
AV		2057	0.70	2.47	5844	8 390 000	19 140[b]
B	Roofing tile	2057	4.09	2.34	4505	4 580 000	13 980[b]
BV		2057	1.14	2.52	4488	5 500 000	18 680[b]
C	Paving brick	2156	2.15	2.35	3925	6 780 000	12 150[b]
CV		2156	0.66	2.42	4974	7 620 000	15 170[b]
D	Drain tile	2057	2.02	2.30	2664	7 010 000	12 420[b]
DV		2057	0.79	2.32	2706	6 520 000	13 530[b]
E	Common brick	2156	9.74	2.11	3148	3 410 000	13 820[c]
EV		2156	7.36	2.18	3550	3 500 000	16 780[c]
F	Salt glazed brick and tile	2156	4.87	1.92	3840	6 080 000	17 610[c]
FV		2156	2.78	1.95	4240	6 290 000	18 340[c]
G	Glazed face brick	2282	6.56	2.10	3306	4 390 000	11 380[c]
GV		2282	5.27	2.21	3812	4 900 000	12 450[c]
H	Sewer pipe	2282	2.68	2.18	2872	4 750 000	14 530[c]
HV		2282	1.90	2.24	3608	7 950 000	18 180[c]

[a] V indicates evacuated body.
[b] Fired at 2003 F.
[c] Fired at 2156 F.

TABLE IV.—PROPERTIES OF PAVING BRICK MADE FROM NORMAL AND DE-AIRED CLAY.

Ware	Weight of 10 Specimens, lb.	Increase in Weight Due to Evacuation, per cent	Absorption, per cent	Rattler Loss, per cent	Improvement Due to Evacuation, per cent
Medium-fired Cleveland ware, unevacuated	86.50	....	2.55	25.43	....
Medium-fired Cleveland ware, evacuated	90.00	4.05	0.70	20.55	19.17
Light-fired Cleveland ware, unevacuated	85.75	....	2.45	21.86	....
Light-fired Cleveland ware, evacuated	89.00	3.25	0.26	16.85	22.97
Medium-fired Canton ware, unevacuated	80.75	....	5.30	22.45	....
Medium-fired Canton ware, evacuated	85.62	6.02	0.20	18.82	19.28

TABLE V.—LOAD TESTS AND ABSORPTION OF STRUCTURAL TILE.

	Crushing Strength, Total Load, lb.		Absorption, per cent	
	Unevacuated	Evacuated	Unevacuated	Evacuated
No. 1	226 000	255 000	6.34	4.39
No. 2	261 000	320 000	6.62	4.51
No. 3	268 000	262 000	6 13	4.91
No. 4	202 000	....	6.00	....
Average	239 250	279 000	6.25	4.60
	Improvement, 25.2 per cent		Decrease, 26.4 per cent	
Net area, sq. in	27.2	25.2		
Net unit strength, lb. per sq. in	8 790	11 100		

Experiences from Commercial Operations Using De-airing:

A well-known paving brick manufacturer reports the following commercial benefits were obtained by de-airing:

> "The improvement in the green column and the green ware by this process was quite striking, even though our previous experimental work had given us considerable indication of what to expect. There was a tremendous increase in plasticity and workability of the shale bar and a noticeable increase in green elasticity and density. The shale ran excellently through the die, with a smooth finish and sharply defined edges and corners.
>
> "We may sum up the improvements in the characteristics of the burned ware as follows: The rattler test abrasion loss was reduced from two to five points. The compression or crushing loads were very greatly increased. As a matter of actual test we have had compression results showing loads as high as 26,000 lb. per sq. in. and we have a whole series running from 18,000 to 25,000 lb. per sq. in. Please note, the crushing strength of granite is frequently given as from 9000 to 35,000 lb. per sq. in., with an average of from 18,000 to 25,000 lb. per sq. in. The absorption tests (5-hr. boiling test made in accordance with the Society's specifications) ran consistently lower at any burning range with the de-aired product than with the unde-aired product."

The first paving brick made by the de-airing process and subject to laboratory tests were several series of 3-in. brick made from Cleveland and Canton shales. Data obtained from tests of these brick are given in Table IV.

Structural tile made by the de-airing process, run out on one of the first commercial de-airing machines, were compared with tile made from the same clay body by normal plant methods. The results are given in Table V.

The stiff-mud de-airing process has also been applied commercially to the manufacture of sewer pipe, drain tile, roofing tile, face brick and refractory brick. Only one installation has been made in the sewer pipe industry to date. An improvement in compressive strength of the de-aired pipe ranging from 10 to 20 per cent has been reported. The body structure as regards visible lamination has also been improved although it is necessary to add that the maximum benefits from the de-airing of sewer pipe have not been obtained as yet. Reports similar to the foregoing are also obtained from manufacturers of drain tile, face brick and refractories. It should also be noted that by reason of the novelty of commercial de-airing and the lack of experience thereby, the possibilities have not been thoroughly explored, and, conversely, there may arise certain unforeseen difficulties and problems in drying and firing that may minimize the possibility of its universal adoption. Such difficulties may prevent some manufacturers from producing de-aired products economically, especially since some raw materials are undoubtedly less susceptible to proper de-airing.

SUMMARY

Summarizing the improvements which may be secured by the commercial application of the evacuation process, the following are the most apparent:

1. Production of stronger, denser, and more uniform quality of ware by the dry-press process.

2. Materials that are now unusable by the stiff-mud process may be worked satisfactorily.

3. Products and types of ware not commercially possible by the stiff-mud process may be made when de-aired.

4. Stronger, less absorbent ware by the stiff-mud process can be secured by firing the de-aired product to the same temperature as used at the present time with normal plant ware.

5. De-aired stiff-mud products will undoubtedly meet specifications regarding quality and properties much more easily than undeaired ware.

DISCUSSION

Mr. G. F. Schlesinger.[1]—Twelve of the largest of the 55 active manufacturers of paving brick in this country have equipped 14 plants with the de-airing process. Considering the economic conditions during the past few years, this is a good indication that the paving brick industry is convinced of the commercial advantages of the de-airing process for manufacturing paving brick.

A questionnaire to 8 of the 12 companies using the de-airing method in 1933 brought forth 7 replies containing the following information:

Through use of the de-airing process 7 companies reported (1) definite decreases in average losses in the rattler which varied from 2 to 6 points, (2) improvement in uniformity of the product, and (3) increases in the proportion of No. 1 brick, passing the A.S.T.M. specifications, which ranged from 2 to 17 per cent. Four of the companies reported that the weight of the de-aired product was 3 to 5 per cent greater than that of the brick produced without de-airing. Six of the 7 replies indicated that the advantages mentioned were secured without additional cost; the seventh reply indicated a slight increase in cost for the de-aired brick.

I have no special comments on the paper by Messrs. Rueckel and Keplinger other than to say that I think the paving brick industry is convinced that the de-airing process is here to stay, and we agree in general with the conclusions of the paper.

Committee C-3 on Brick has instructed its subcommittee on paving brick to make a study of de-airing with a possible resulting recommendation of changes in the A.S.T.M. test for paving brick.

Mr. W. C. Rueckel[2] (*author's closure by letter*).—Mr. Schlesinger's remarks clearly indicate (and so also does all the information we have obtained in our work) that the majority of clay plants will install de-airing equipment mainly to effect a considerable saving in operating costs by an increased production of first quality brick.

Data given in the paper show that the de-aired product will have a much lower absorption value and an altered surface texture. The alterations of these properties will undoubtedly affect the bond between brick and mortar to no slight degree. The necessity for an investigation of the bond between mortar and brick in which de-aired brick are used with typical mortars is therefore apparent.

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[1] Chief Engineer and Managing Director, National Paving Brick Assn., Washington, D. C.

[2] Research Engineer, Engineering Experiment Station, Ohio State University, Columbus, Ohio.

METHOD AND APPARATUS FOR DETERMINING THE INTERVAL REQUIRED TO DISPERSE OIL COLORS THROUGHOUT A PAINT MEDIUM

By M. Rea Paul[1] and George Diehlman[1]

Synopsis

A necessary property of colored pigments ground in oil is that they rapidly disperse when mixed with a paint medium for tinting purposes.

An apparatus and comparative method is described for determining the time interval (in seconds) required to disperse colored pigments ground in oil throughout a white paint medium. The specific method is that colored pigment in oil, white paint and lead shot of defined size, all contained in a special glass bottle, are agitated in a specified manner. Uniform distribution and mixing are readily obtained. The total interval of agitation is carefully timed, the end point taken when by visual observation homogeneity or absence of streaking is secured. Results so obtained have been found comparable to practical mixing experience. Two tables are included, giving typical results obtained by the method.

Introduction

One of the necessary properties of colored pigments ground in oil is rapid dispersion when mixed with a paint medium for tinting purposes. At the present time no known method, other than practical mixing by hand, is available for determining with any accuracy the time interval required for this dispersion to be completed. After considerable experimentation with various means that could be employed to achieve agitation that involved stirring, shaking and rotating, etc., a satisfactory selection was made.

Herein is described a method with apparatus for comparatively determining time in seconds necessary for complete dispersion of colored pigments in oil throughout a white paint medium. While there may be other possible uses for this method, work with it has so far been restricted to paste colors-in-oil.

All dispersion data given in this paper were obtained with white lead paint mediums, but paints made from other white pigments have been used with equal satisfaction where the consistency was somewhat similar to that given in Table I. Consistency of the white paint medium is specified because for the purpose intended, it facili-

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[1] Research Laboratories, National Lead Co., Brooklyn, N. Y.

tates rapid and uniform final dispersion with the stated conditions of procedure.

We believe, however, that by varying the size and combinations of the lead shot, the method can be applied to a wider range of comparative dispersive problems. The lead shot variations would necessarily have to be developed for each individual case because of differences in materials and viscosities. Possible applications that might be made the basis for further study involve comparative dispersive intervals for pastes generally. In the case of whites, the color-in-oil could be used as an indicator. By reversal, using a stock paste and a colored indicator, the dispersion interval for various vehicles with reference to the paste might be determined.

APPARATUS AND EQUIPMENT

Glass Mixing Bottle.—The dispersion test is conducted in a specially constructed tubular glass bottle, a sketch of which, with dimensions, is shown in Fig. 1. The bottle is stoppered with a cork, concaved at the bottom and inserted just to the upper end of the shoulders, thus duplicating the opposite rounded end of the bottle. The neck and shoulders of the bottle are covered on the outside by means of glued black paper, thus shielding this interior portion from view. Shielding removes an objectionable contrast introduced when the stopper can be seen during actual operation.

TABLE I.—MOBILITY DETERMINATION AT 21 C.[a]

Time in seconds for the plunger to fall through 10 cm. of the material using the disk with 50 holes.

TOTAL LOAD, G.	TIME, SECONDS
68	28.0
100	9.0
150	4.0
200	2.5

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[a] For descriptive details of apparatus and manipulation, see H. A. Gardner, "Physical and Chemical Examinations of Paints, Varnishes, Lacquers and Colors," Sixth Edition, p. 521 (1933).

Holder for Mixing Tube.—The holder is made of wood, painted black. The dimensions are: height 28.0 cm., width 11.0 cm., thickness 3.0 cm. The center of this board is cut out and shaped to allow for insertion of the glass mixing tube to about one-third of its diameter. A copper strap $\frac{3}{4}$ in. wide, which can be made tight against the mixing tube by means of a winged nut and bolt, is attached to the board. For attaching this holder to the rotating shaft, a $\frac{1}{4}$-in. standard pipe flange is fastened to the back at a point 23.4 cm. from the top to the flange center. The rotating shaft was made from a piece of iron rod threaded at one end and sprung to give an angle of 12 deg. from the vertical (see Fig. 2).

Drive and Rotating Device.—Any suitable drive and rotating device can be used provided the shaft connected to the holder is turned at 27 r.p.m.

Measuring Tube for Colored Pigments in Oil.—For comparative purposes, it is essential that equal volumes of the colors-in-oil be charged. Where the specific gravity is known in each case, the charges may be weighed directly into the mixing tube after suitable adjustment by calculation.

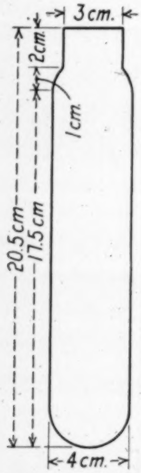

FIG. 1.—Glass Mixing Bottle (Inside Dimensions).

For simplification, a constant volume measuring tube was adopted made up as follows: glass tube 20.0 cm. long with flat ends, inside diameter 0.5 cm. graduated from one end to hold 3.0 ml. at 21 C. Into this tube is inserted a snugly fitted glass plunger tapered to a point at one end. The color-in-oil is sucked into the tube to the mark and discharged by means of the plunger. By this procedure, reasonably accurate volumes are quickly measured.

Materials Required:

(*a*) Supply of No. 11 lead shot (approximately 0.06 in. in diameter).

(*b*) Standard white paint. For experimental purposes, regular flat white lead paint was employed, after it had been reduced with turpentine to the approximate consistency given in Table I.

TEST METHOD

Loading.—The mixing bottle shown in Fig. 1 is counter-balanced in an upright position by means of a suitable holder and the following additions introduced in the manner and quantity specified: 550 g. No. 11 lead shot (approximately 0.06 in. in diameter), 3 ml. color-in-oil (recounter-balance) and 75 ml. white paint (by weight).[1] For good initial distribution of the color, it is essential that the additions be made in the sequence specified. All standard white paint that will be required should be carefully strained and well stirred prior to weighing each charge. When charging color-in-oil, care should be taken to prevent contact with the glass walls of the mixing tube as much as possible until the walls have been wetted with white paint. This wetting is accomplished by tilting the bottle at an angle and slowly turning. The bottle is then stoppered with the special cork and fastened in the holder as shown in Fig. 2. When the charge is prepared, it should be run immediately, as standing will in time permit certain colors-in-oil to seep through the shot, thus providing a slight dispersive advantage.

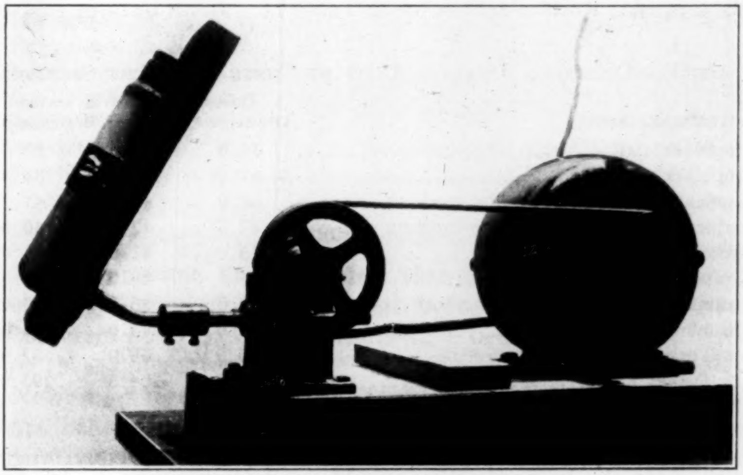

FIG. 2.—Apparatus with Mixing Tube in Place.

Rotating.—Rotation is started simultaneously with timing (stop watch). The lead shot is distributed by gravitational force from one end of the bottle to the other, dispersing the color-in-oil throughout the paint. The angle of 12 deg. at which the holder and mixing bottle is set, concentrates the impact against the walls, thus removing any adhered color-in-oil at these parts.

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[1] In the case of flat white lead paint, 75 ml. weighs 195 g. approximately.

Reading.—When homogeneity, or obliteration of streaking, is noted through visual observation, the end point has been reached. The timing should cease simultaneously with notation of the end point and the lapse of time in seconds taken as the reading.

DATA AND DISCUSSION

With the experience of a few trials with this method, different operators can determine the end point sharply enough to provide reasonable checks. The degree of accuracy obtainable with the described method was tested by having three operators separately determine the dispersion rate of similar colors-in-oil, each of a different brand. In each test the same standard white paint was used from a bulk supply. Results are shown in Table II. In all but one instance satisfactory checks were secured. The exception (last sample of raw Turkey umber) was of stiff paste consistency and of a type that in itself is often incompletely dispersed. This was purposely included to show the limitations of the method as at present applied. When tested the bulk of such colors-in-oil disperse throughout the white paint medium fairly rapidly but are followed by a breaking out of small specks of color at irregular intervals. Such colors require considerable time before homogeneity is obtained, and as a result an approximate, instead of a clearly defined end point, is noted. This approximate end point may be obtained by observing the elapsed seconds when homogeneity in the mixing bottle is first observed but allowing the stop watch to continue running. Successive noted time interval readings are made after the disappearance of each outbreak of specks and by this process the end point is finally reached.

TABLE II.—DISPERSION INTERVAL TESTS BY THREE DIFFERENT OPERATORS.

	DISPERSION INTERVAL, SECONDS		
COLOR-IN-OIL[a]	OPERATOR A	OPERATOR B	OPERATOR C
Lampblack	53.0	58.0	52.0
Lampblack	57.0	52.0	54.0
Chemically pure chrome yellow-medium	44.0	45.0	41.0
Chemically pure chrome yellow-medium	42.5	42.0	40.0
Chrome green medium	45.0	41.0	44.0
Chrome green medium	45.5	40.0	43.0
Ultramarine blue	38.0	36.0	33.0
Ultramarine blue	47.0	43.0	44.0
Raw Turkey umber	36.0	38.0	37.0
Raw Turkey umber	231.0	204.5	293.0

[a] Each sample of a different brand.

Colors-in-oil suitable for tinting purposes as made by different manufacturers vary considerably in dispersability. Several pairs of similar colors each from a different source were tested by the method described and the results given below. Chemically pure Prussian blue is particularly outstanding and is among the most difficult colors to disperse:

DISPERSION INTERVAL OF COLORS-IN-OIL OBTAINED FROM DIFFERENT SOURCES.

COLOR-IN-OIL	DISPERSION INTERVAL, SECONDS
Chemically pure Prussian blue	305.0
Chemically pure Prussian blue	147.0
French ocher	40.5
French ocher	27.0
Raw sienna	32.5
Raw sienna	44.0
Burnt sienna	41.0
Burnt sienna	99.0
Van Dyke brown	45.0
Van Dyke brown	37.0
Drop black	41.0
Drop black	36.0

CONCLUSIONS

It has for some time been felt that considerable variation in dispersability unquestionably existed between similar colors-in-oil from different sources, but no means have been available for determining these differences.

Now, after tests and practical trials with different operators, the authors believe that a satisfactory method has been evolved for comparatively determining the speed at which colors-in-oil added for tinting purposes can be dispersed throughout a white paint medium.

From the information developed in this paper it is apparent that an appreciable variation does occur that is now measurable. The authors realize that the apparatus as described is subject to improvement and present it, together with the method, in the hope of receiving constructive comments that will stimulate further thought along these lines.

DISCUSSION

Mr. J. J. Rankin.[1]—I should like to ask whether the fineness of the pigment has anything to do with the rate of dispersion.

Mr. George Diehlman.[2]—Not necessarily in the present application of the method. We have dealt with the comparative dispersion rates of colored pigments in oil throughout a paint medium and therefore the particular vehicle in which the colored pigment is ground as well as the consistency of the paint medium become influential factors.

Mr. Rankin.—What would be the effect then of various type iron oxides as to rate of dispersion?

Mr. Diehlman.—We have covered to some extent in this paper the dispersion rate of different iron oxide pigments in oil, for instance, French ocher as compared with raw sienna, etc. If reference is being made to the possibility of applying this apparatus to show differences in fineness of dry pigments, I might say that we are contemplating additional work of this type where the comparative degree of fineness will be obtained from the dispersive rate in a common, neutral good-wetting vehicle.

Mr. G. E. Walker.[3]—That 27 r.p.m. is quite interesting.

Mr. Diehlman.—The 27 r.p.m., the 12-deg. set of the mixing tube holder, the size and amount of lead shot, all required considerable trial before adoption. The degree of set was established by introducing lead shot and clear fairly viscous mineral oil into the glass mixing tube, placing this in the holder set in a vertical position and then rotating slowly enough to observe if uniform distribution of the shot was being obtained. When the 12-deg. set accomplished this purpose, both paint and color in oil were tried and by repeated trial the speed of rotation and the size and quantity of lead shot required for uniform distribution were obtained.

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[1] Production Engineer, St. Joseph Lead Co., Josephtown, Pa.

[2] Research Laboratories, National Lead Co., Brooklyn, N. Y.

[3] Technical Director, Southern Mineral Products Corp., Piney River, Va.

A DISCUSSION OF THE ACCURACY AND UTILITY OF METHODS OF TEST FOR MASS TONE AND TINTING STRENGTH OF DRY COLORS

By J. W. Ayers[1]

Synopsis

A survey is made of the Society's proposed Tentative Method of Test for Mass Color and Tinting Strength of Dry Color Pigments or Pastes[2] and several changes in apparatus, methods, and materials are recommended. The conclusions include changes as suggested by a number of color laboratories.

Experimental data include the testing of several typical dry earth colors with a study of the effect of variations in testing conditions. Photo-electric spectrophotometric equipment was used for brightness and color measurements in the determination of the effect of the number of grinding mulls and the consistency of the test paste on color and tinting strength. Properties of representative plant grinds of the same pigments were compared with the test data to ascertain the testing conditions most closely approximating practical plant conditions. Curves and tables are given to illustrate the experimental data, and to demonstrate that testing conditions must be closely controlled to obtain duplicate results.

It is suggested that a similar study be made of all groups of dry earth colors before definite testing requirements be approved.

Introduction

Any acceptable routine testing procedures for mass color and tinting strength must be so designed that unskilled operators can check each other within a reasonable degree of accuracy. The test should also require a minimum amount of time. Any procedure which might show variations should be eliminated and short cuts, even at the sacrifice of extreme accuracy, seem desirable.

It is not proposed that the Society's proposed Tentative Methods of Test for Mass Color and Tinting Strength of Dry Color Pigments or Pastes (D 387), being recommended this year by Committee D-1,[2] should be used in evaluating any but the same type of colors. It is operative only with those colors of approximately the same chemical composition, saturation, hue, and brightness. The relative quality of colors having widely different properties require methods of test completely beyond the scope of these tentative methods.

In the following discussion, an attempt is made to show the inaccuracies and inconsistencies of the methods under consideration.

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[1] Director of Research, C. K. Williams and Co., Easton, Pa.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 871 (1934).

Methods for improving the present procedures in both accuracy and speed are suggested.

APPARATUS AND MATERIALS RECOMMENDED

Apparatus:

The Balance.—The tentative method as modified requires a regulation laboratory balance. No sensitivity is proposed. J. D. Todd of the Sherwin-Williams Co. suggests that the sensitivity of the balance be to at least 1 mg.

Grinding Instruments.—The texture, fineness, and consistency of the natural earth pigments are important to both color manufacturers and consumer. An experienced operator, using a spatula, is capable of making a quick, approximate appraisal of the properties of texture, fineness and consistency. In such instances, the spatula serves a very useful purpose but it cannot be recommended for general use because much experience is essential before it can be handled effectively.

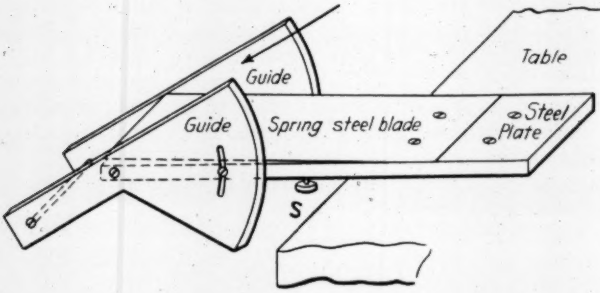


FIG. 1.—Proposed Scraper.

Rubbing Surface.—The texture of the rubbing surface apparently may vary considerably without affecting, materially, the accuracy of the test. As long as the two samples under test are both rubbed on the same surface, the results should be comparable. A series of tests were run to show differences in tinting strength development on smooth and newly ground glass surfaces. In no case was the difference greater than 2 per cent.

French Scraper.—Mr. L. J. Venuto of the Binney & Smith Co. has invented a scraping device which does away with the objections to the French scraper. A sketch of this apparatus is shown in Fig. 1.

The instrument consists essentially of a rigid steel plate to which is attached a spring steel blade as indicated in the drawing. Guides are attached to the sides, their function being to guide the glass slide or tin plate against the spring blade which is maintained to a constant pressure by means of adjusting screws. Venuto reports several years' experience with this instrument and claims his results to be entirely satisfactory. It has a number of attractive features and overcomes inaccuracies in the present procedure and is, therefore, worthy of serious consideration in place of the French scraper now recommended.

Sheets of Flat Glass or Flexible Celluloid.—Thin gage stainless steel or nickel, 3 in. square, with one corner turned up for ease of handling, would seem more durable than glass or celluloid.

Materials:

Linseed Oil.—Heat-bodied oils are to be avoided because of excessive strength development of colors of hard texture because of the high consistency of such oils.

Zinc Oxide Paste.—A. F. Brown, of the Imperial Color Works, in his comments on the tentative specifications, states that although the pastes recommended work well, the laboratories of his company have found a paste of 25 parts of heat-bodied oil of "O" Litho varnish consistency and 100 parts of Green Seal zinc oxide by weight to be more simple to prepare, and there is no separation of pigment and oil on aging.

RECOMMENDED PROCEDURES FOR MASS COLOR AND TINTING STRENGTH

Mass Color:

In the schedule for pick-ups, a pigment demanding 50 rubs is to be picked up after each 15 rubs. Since 50 is not divisible by 15, an odd number of 5 rubs remains. Therefore, 60 mulls, which is divisible by either 15 or 20, is recommended.

In a letter to the author the questionable value of the test for floating and bronzing is pointed out by A. F. Brown. The behavior of this phenomena is dependent on the type of vehicle used. This test pertains to a particular application of any color and seems to be out of place in this tentative specification. It is recommended that it be dropped.

Commercial paste colors very often contain combinations of heavy bodied linseed, soya bean, poppy, corn, tung and linseed oils. Colors ground in such combinations flood excessively. This flooding renders any mass color determination totally unreliable and inaccurate.

Tinting Strength:

The present schedule for the reduction of high-strength carbon black, lampblack, and ordinary carbon black calls for a reduction of 20 to 1. These pigments have very high tinctorial value and the accuracy of the tinting strength determinations of these pigments would be greatly increased if the reductions were made at 50 to 1 instead of 20 to 1. The brightness of a 20 to 1 reduction of these blacks is very low and the saturation high, making observation of small differences questionable.

The evaporation of the solvent, when Japan colors are subjected to the atmosphere, makes impossible the accurate weighing of such colors containing solvent. Inaccuracies are thus injected into the tinting strength determination rendering it unreliable.

EXPERIMENTAL DATA

Apparatus and Procedure:

For the rub-up of pigments, the A.S.T.M. recommendations were followed. A ground glass slab, 3-in. muller, refined linseed oil, Brown burette, zinc oxide paste, etc., were employed in the testing. An unweighted muller was used with a pressure of 10 lb. applied by the operator. Let-down tests were weighed on a flat glass, and mixed on the same glass to avoid loss.

For the determination of brightness of let-downs, a photo-electric photometer was employed. The assembly consisted of a light source, color filter to correct to the visual spectrum, and an American Photoelectric Corp. photo-electric cell using two stages of amplification. The light source was hemispherical, and arranged so that two samples —the standard and the sample under observation—could be shifted into place for quick comparative readings. The correction filter was a solution of potassium dichromate and copper sulfate, in such proportions as to correct the particular photo-electric cell to the same sensitivity as the eye.

For color analysis, an assembly similar to the above was used with the substitution of a spectrometer for the filter. This enabled the experimenter to measure reflection at any specified wave length of light.

In brightness determinations, a particular sample was chosen as standard and all others in the test series referred to this standard. In color analysis, a magnesium carbonate block was used as the standard.

All measurements were made of the paste surface and *not* through glass.

Plant versus Muller Grinding:

Some knowledge of the ease with which any color can be dispersed in oil and the susceptibility of any color to grinding energy is helpful in evaluating that color. Some relation between the tinctorial development of a color by practical plant grinding and tinctorial development under the muller is desirable. To establish this relationship experimentally, samples of colors in oil were procured from four leading paint manufacturers. Of the many samples tested, four have been chosen to illustrate the relation between plant grinding and muller grinding. The plant production data on these samples are given in Table I.

Effect of Mulling on Tinting Strength and Comparison with Mill Grinds:

To determine the effect of mulling on tinting strength, umbers A and B, and Venetian reds A and B were mulled from 0 to 300 times and the tinting strength determined by measurement of brightness of the sample, and a comparison of the brightness figure was made. The results of these tests are shown in Fig. 2.

TABLE I.—PLANT PRODUCTION DATA.

	Condition	Plant Paste Sample		Oil Absorption	Mill	Rate of Production, gal. per hr.
		Pigment	Oil			
Burnt umber A	Dry	...	.....	62.04	....	..
	Paste	100	92	.....	Stone 36 in.	15
Burnt umber B	Dry	...	.....	48.84	....	..
	Paste	100	97.2	.....	Stone 26 in.	8
Venetian red A	Dry	...	.....	22.44	....	..
	Paste	100	25	.....	Stone 30 in.	20
Venetian red B	Dry	...	.....	23.76	....	..
	Paste	100	38.85	.....	Stone 26 in.	20

The let-down in zinc oxide paste was 20 to 1, and the pigment-oil ratio the same as the plant samples above. It will be noted that the plant samples are equivalent in tinting strength to the following number of mulls:

	MULLS EQUIVALENT TO PLANT GRIND
Umber A	25
Umber B	60
Venetian red A	15
Venetian red B	25

With the exception of umber B, the plant grind was far inferior to that obtained by a minimum of mulling. It may be that the pigment-oil ratio information supplied with umber B was incorrect. It will be noted that 60 mulls approached the point of ultimate strength development, while above 100 mulls there is very little further development.

In all these tests the 60-mull sample was considered to be 100 per cent tinting strength and all other samples compared to it.

A comparison of the Venetian red graph with the burnt umber graph in Fig. 2 shows that the natural earth color responds in tinctorial development to a greater degree than the synthetic Venetian red. The tinting strength of the umber increases from 40 to 110 per cent or 70 points with 100 mulls while the Venetian red increases from 60 to 100 per cent or 40 points with 100 mulls. The texture of these two pigments is very different. The umber is relatively large, irregular, and harsh, while Venetian red is fine, uniform, and soft.

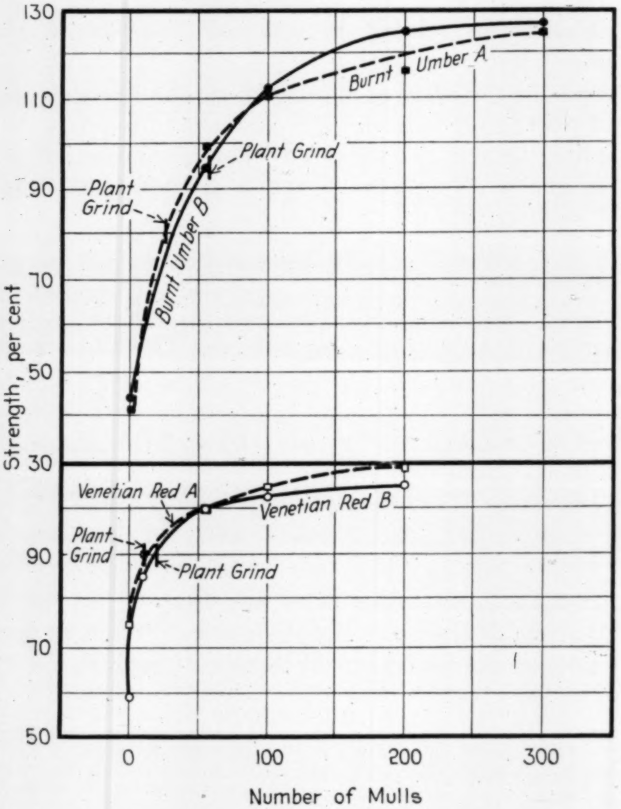
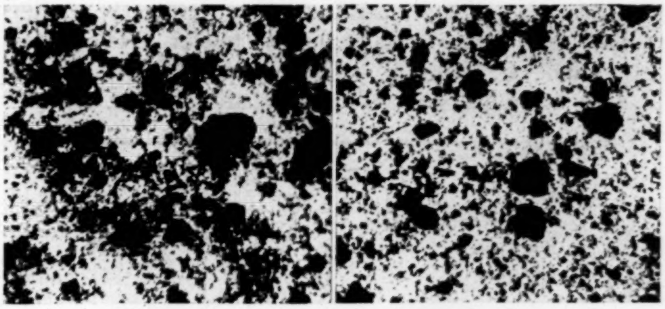
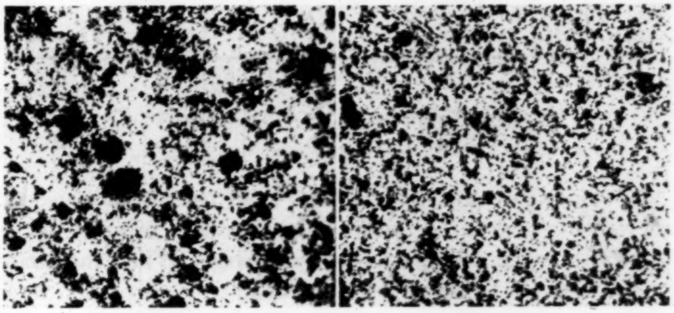


FIG. 2.—Effect of Mulling on Strength.

The agglomeration and particle size distribution of paste samples of burnt umber B with no mulling, 60 mulls and 300 mulls as compared to a plant grind of the same color is shown by the photomicrographs in Fig. 3. The particle dispersion and agglomerate breakdown of the plant grind sample appears to be equivalent in dispersion and breakdown to the color paste mulled 60 times. The curve for burnt umber B, Fig. 2 shows the tinting strength of the burnt umber B plant grind to equal a 60-mull grind of the dry burnt umber B in oil. The photomicrographs therefore check the tinting strength tests with dispersion. It should be observed that the burnt umber B dry sample mixed in oil with no mulling shows very poor dispersion, whereas the same sample mulled 300 times shows excellent dispersion.



No mulling. Plant grind.



60 mulls. 300 mulls.

FIG. 3.—Effect of Mulling on Burnt Umber B Paste Samples 5 μ Thick (×200).

A. F. Brown of Imperial Color Works, L. W. Ryan of United Color and Pigments, and Seviere Bonnie of Kentucky Color Works, report that the number of mulls specified in the rub-out schedule of the tentative specifications represent approximately the grinding which the chemical colors get in practical plant production. J. A. Todd of Sherwin-Williams Co. reports 200 mulls of an American vermillion equivalent to a plant grind. It would be well if these inconsistencies were checked experimentally. Standard Ultramarine Co. reports the tentative specifications as drawn for ultramarine blues to be entirely satisfactory.

Effect of Consistency on Tinting Strength:

Tests were made with umbers A and B and Venetian red B to determine the effect of consistency of the paste under a muller on the tinting strength developed. In each series, the pastes were tested from as heavy a paste as it was possible to work under the mullers to a paste so thin that it ran from under the muller. The latter paste may be described as "soupy." The pigment-oil ratio as shown in Table I, with 60 mulls, was set up as a standard of 100 per cent.

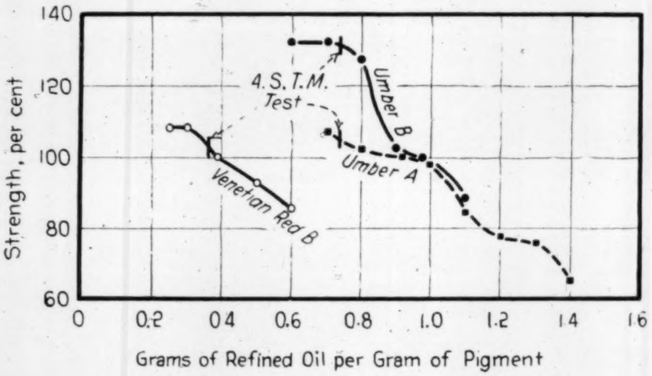


FIG. 4.—Effect of Consistency on Strength.

When the amount of oil used was less than the amount used in the standard pigment paste, an additional portion to bring it up to that quantity was mixed in the paste after mulling. When the amount of oil was greater than the amount in the standard, a correction was made in weighing the pigment paste; thus, the same amount of dry pigment was present. The results are graphically shown in Fig. 4.

In each case, the maximum strength was developed with a very heavy consistency paste. There appears to be a definite point of consistency at which maximum grinding action takes place, and beyond this, higher consistency does no further good. In the case of umber A this point is 0.7 g. of oil per gram of pigment, umber B 0.7 g. of oil per gram of pigment, and Venetian red B 0.3 g. of oil per gram of pigment.

The ideal conditions for testing would be in the flat range of each curve. Slight differences in consistency would then not inject an error into the test. Unfortunately consistencies in this range are heavier than can well be worked under the muller. The pigment-oil ratios recommended in the proposed tentative methods which work well under the muller come as close to the ideal as possible. These points are on the steep slope of each curve and show the necessity of working at equal consistency for accuracy. Some simple and workable method of actually measuring the consistency of the paste to be tested would be a real contribution to the specifications. The iron oxides and earth colors vary more in oil absorption than any other classification of colors. Large errors can be injected into the mass color and tinting strength results unless adjustments to equal consistency are made for variations in oil absorption.

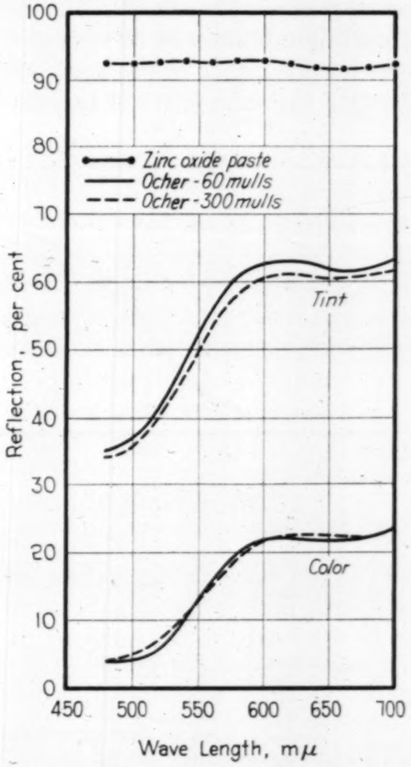


FIG. 5.—Effect of Mulling on Color and Tint.

Effect of Mulling on Color and Tint:

To test the effect of excessive mulling on color and tint, a domestic ocher was chosen as the test pigment. This material was mulled 60 and 300 times using the quantities of 1 g. pigment and 0.55 g. oil. The color change was measured spectrophotometrically as indicated on the two lower curves of Fig. 5. It will be noted that excessive mulling decreases the percentage of true yellow, but increases that of red and green. This indicates a muddying of the color, a fact known to color testers.

The color pastes were mixed with zinc oxide paste in a 10 to 1 let-down and the color measured as above. The material mulled 300 times is naturally less bright because of its increased tinting strength, but it is also proportionally higher in the green and red portions of the spectrum, indicating the same effect of excessive mulling on tint as on color.

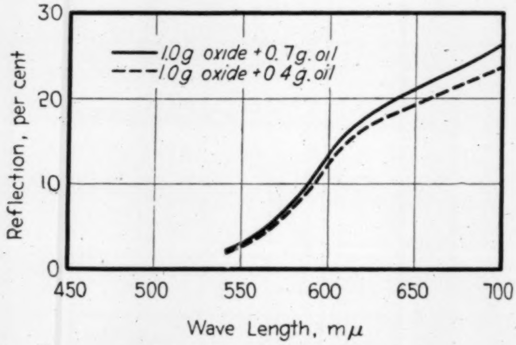


Fig. 6.—Effect of Consistency on Color.

The contour of the tint curve in Fig. 5 does not conform exactly with the color curve, particularly in the range between 575 and 650 mμ. This difference can be attributed to the influence of the zinc oxide in the tinting paste as the brightness of the zinc oxide in this range is at its highest point.

The upper curve is merely that of the zinc oxide paste for comparison. Because of the low opacity of the zinc oxide paste, it was necessary to back the sample with a magnesium carbonate block which is the reason for the brightness of the zinc oxide being higher than normal.

Effect of Consistency on Color:

A light red pure iron oxide was used to test the effect of consistency on color. This material was mulled 60 times with 0.4 and 0.7 g. of refined oil and a color analysis made of each sample. With a smaller quantity of oil, it will be seen, as shown in Fig. 6, that the brightness is lower by a considerable degree.

The above change is found, with change in consistency, when almost any color is tested. In the case of blacks, the heavy consistency pastes cause the mass color to appear gray. The thinner the black paste, the more dense the mass color.

At the peak of the curve in Fig. 6, that is, at 700 $m\mu$, the low consistency paste shows a brightness of 26 per cent; the high consistency paste, a brightness of 23 per cent, the difference being 3 per cent. Assuming that 26 per cent is the maximum brightness of the sample, the actual difference in brightness due to change in consistency is $\frac{3}{26}$ or 11.52 per cent.

CONCLUSIONS

1. The balance recommended should have a specified sensitivity of 1 mg.

2. For general testing, the muller is recommended rather than the spatula.

3. The texture of the rubbing surface has little, if any, bearing on the results of mass color and tinting strength tests.

4. A new smoothing and leveling device, to take the place of the French scraper, is proposed by L. J. Venuto.

5. Nickel or stainless steel weighing plates are recommended in place of glass or celluloid.

6. Heat-bodied oils are to be avoided in mass color and tinting strength tests because of high consistency.

7. A zinc oxide paste of 25 parts heat-bodied linseed oil of "O" Litho varnish consistency and 100 parts of Green Seal zinc oxide is proposed in place of the paste formulations given in the tentative methods.

8. In the schedule for pick-ups, those pigments requiring only 50 mulls should be given 60 mulls to avoid an odd number of mulls, if intervals of 15 mulls are specified for each pick-up.

9. The floating and bronzing test recommended seems to be out of the scope of the tentative methods and should be dropped.

10. Because commercial pastes are formulated with a mixture of drying and semi-drying oils, which cause flooding of the color, mass color tests on these pastes are inaccurate.

11. For greater accuracy, the let-down of the high-strength carbon pigments should be 50 to 1 rather than 20 to 1 as recommended in the tentative methods.

12. In most cases the tinting strength development of the iron oxides and mineral earth colors with 60 mulls is greater than the tinting strength development of the plant grinds of these colors.

13. One hundred mulls of the iron oxides and mineral earth colors give almost optimum tinting strength development.

14. The natural earth colors respond to the grinding action of the muller to a greater degree than do the synthetic iron oxides.

15. The photomicrographs showing the dispersing action of the muller check the curves showing the increase in tinting strength with increased number of mulls.

16. High-consistency pastes develop tinting strength under the muller more rapidly than low-consistency pastes. All tests for tinting strength should be run at the same or equal consistencies. Adjustments should be made for similar colors having widely different oil absorptions.

17. Japan colors contain volatile matter, the evaporation of which makes accurate weighing impossible. Consequently tinting strength tests of these colors by the recommended procedure in the tentative methods would be unreliable.

18. Excessive mulling will change the mass color and tinting hue of many iron oxide colors and some chemical colors.

19. Although all chemical color producers are not agreed that the proposed schedule of rub-up approximately compares with plant grinding, the general consensus of opinion is that it does.

20. Almost all colors become less brilliant and consequently darker in mass color as the concentration of the color increases in a given amount of oil. Black pigments become more dense as the pigment concentration is decreased in a given quantity of oil. The above statements are particularly true of the high pigment - low oil ratios. All colors to be tested for mass color should be run at the same or equal consistency.

21. The above discussion and experimental data serve to show the many variables encountered in testing colors. A thorough investigation of the behavior of all types of colors to these variables will be necessary before practical and workable specifications can be justly approved.

Acknowledgment.—The author wishes to acknowledge the assistance of Messrs. Fred VanDoorninck, W. J. Latimore, and C. H. Love in preparing the above data and discussion.

DISCUSSION

Mr. A. F. Brown.[1]—In some of the industries, particularly the printing ink industry, heat-bodied oil is used exclusively in testing color, because it is the principal vehicle used in manufacture, so I do not agree with Mr. Ayers' suggestion that heat-bodied oil not be used—I think it would have to be used because it has quite a lot to do with the apparent shade.

In view of the fact that the Society is interested in having its methods generally applicable and inasmuch as the printing ink industry is a big consumer of color pigment, there should be additions to these specifications outlining general methods of testing that would be satisfactory to the printing ink industry as well as to the paint industry. Of course the printing ink man has in mind the application of an infinitely thin film to a sheet of paper and very often the color effect of the same color will be entirely different if drawn out on paper as compared with the pastes on a slide. In our laboratories, in testing a color to be used in printing ink, we draw down a very thin film on paper, and heat-bodied oil is used as the vehicle. I think before this proposed method is adopted another column should be added indicating the amount of color and "varnish" required to make these different inks so that the method may be applicable to printing inks as well as to paint.

Mr. R. D. Bonney.[2]—In establishing specifications, I believe that sometimes we overlook the fact that materials for which we are drawing specifications are not used exclusively in the paint industry or in the varnish industry. I believe that in the work of Committee D-1 it should be recognized that the materials with which we are working are used for a number of different purposes other than for making colors in oil or mixed paint. In the case of pigment, different vehicles give entirely different results on tests used in specification work. I believe the committee should recognize that for different industries, different types of test are necessary. It would be well worth while to attempt to work out satisfactory testing methods for the various uses to which the products are to be put rather than having constantly in mind the drawing up of requirements for paint uses alone.

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[1] General Manager, Imperial Color Works, Glens Falls, N. Y.

[2] Director of Research, Congoleum-Nairn, Inc., Kearny, N. J.

MR. BROWN.—Our experience indicates that probably 80 or 85 per cent of the total color consumption of the country, excluding black and white, is used by the paint and printing ink industries, using the term paint in a very general sense to mean industries that will test the same as the paint industry—the linoleum industry, for example. If an attempt is made to draw up specifications for some of the smaller consumers, it will be an almost endless job. We are constantly confronted with that problem; we have certain customers who use a color in a particular manner, so we assign a special number to those colors and outline the tests that that particular customer requires. For example, if a man is going to use a color for crayon instead of rubbing it in the oil and drawing it out on paper, as we would do for printing ink, we make a small wax crayon and then we make one with the standard color, and see if they "write" the same. For carbon paper consumers we make a carbon paper on a small scale, etc. I doubt very much if it is feasible for the Society to provide for all those different industries, but it is something that might be worth while. The printing ink manufacturers are now drawing up a specification for themselves along the same general lines as that for the paint industry, because there was none that could be considered standard. I know of no other industry that has drawn up any special specifications, but we do have to make up vehicles from dextrine, starch, gum arabic, etc., for particular customers, because different vehicles give different effects.

MR. R. H. SAWYER.[1]—A question of detail—how were the 5-μ sections for the photomicrographs secured?

MR. W. J. LATIMORE.[2]—The sample of the paste as mulled up was taken and a small daub covering an equal area in each case was placed on a slide. A cover glass was placed over the daub. Then, by taking a thin strip of rubber or a piece of inner tube and wrapping it around the index finger, the cover glass was pressed down and the daub squeezed out. This gave a smear that was wedge shaped, the thinnest portion being in the center. The slide was then placed in the microscope and the depth measured by focusing until an area 5 μ in depth was found. The photomicrograph was made at this point.

MR. G. F. A. STUTZ.[3]—I should like to have Mr. Love elaborate on conclusion No. 16 of the paper which states that "All tests for tinting strength should be run at the same or equal consistencies." I wonder if he would indicate what the tolerance is?

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[1] Chemist, Krebs Pigment and Color Corp., Newport, Del.

[2] C. K. Williams and Co., Easton, Pa.

[3] Chief, Pigment Research Division, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

MR. C. H. LOVE.[1]—In the dry color field, pigments are encountered which vary widely in oil absorption. In such cases it would be necessary to make a preliminary oil-pigment rub-out to determine the correct working consistency, and then to proceed with the standard test. In cases where the same pigment would be tested from day to day for manufacturing control or shipment, the pigment and oil would each be weighed or measured, and the test made as specified without adjustment. In the paper were mentioned two pure iron oxides, one of the precipitated and one of the calcined type. The first has an oil absorption of between 65 and 85 and the second, between 20 and 30. If not adjusted to nearly equal consistencies the tests on these two pigments would not be comparable for either color or strength.

MR. STUTZ.—In case the tables given were somewhat enlarged to include the two types of iron oxide, or to include any other pigment, would that point of widely differing consistencies be met?

MR. LOVE.—I believe it would, and in the work we are carrying out we aim to cover all the different pigment groups and eventually to present these data. We have already determined that on a number of pigments the number of mulls should be reduced from 100 to 60, and we plan to determine the correct figure for all pigment classes.

MR. A. E. STAUDERMAN.[2]—In conclusion No. 7, I notice that a zinc oxide paste of twenty-five parts heat-bodied linseed oil of "O" Litho varnish consistency and 100 parts of Green Seal zinc oxide, is recommended instead of the paste formulations specified in the Society's method. Formulas for both types were given and it is merely a question of which is wanted. It has been our experience that paste made with all heat-bodied oil or blown oil of any type makes stringy paste, which makes for considerable difficulty in weighing without stringing over the balance pan. We prefer the short buttery paste for that reason.

MR. BROWN.—In our own laboratories we use the formula suggested, twenty-five parts of heat-bodied linseed oil of "O" Litho varnish consistency and one hundred parts of Green Seal zinc oxide; we do not have the trouble Mr. Stauderman spoke of, possibly for the reason that, in view of the large amount used in our routine work, when we grind the zinc oxide, we put it in tubes and squeeze it out just like one would tooth-paste. Possibly if we were taking it out of a wide-mouthed bottle or can, we might have difficulty. We found that if we ground it in linseed oil, such as raw or refined, we had a separation after a few weeks. We do not have that separation with the heat-bodied oil. The other formula contemplated, which includes the use of soap, is perfectly all right. But in drawing up specifications, materials that can be found in every laboratory or that are easily obtained should be used, and heat-bodied oil is easily obtained.

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[1] Chief Research Chemist, C. K. Williams and Co., Easton, Pa.

[2] Director of Laboratories, Devoe & Raynolds Co., Brooklyn, N. Y.

MR. H. A. NELSON.[1]—Referring once more to the zinc oxide pastes. Would the difference in dispersion, or the fact that there is better dispersion in the case of heat-bodied oil in any way affect the number of mulls required to obtain a certain tinting strength?

MR. BROWN.—I do not think so, at least not with precipitated pigments.

The thing to keep in mind in all this testing is not the theoretical tinting strength of a color, but the practical tinting strength. It really disturbs me greatly when I go into paint factories and see the half grinds many of these expensive colors get. I have taken samples where 50 per cent of the tinting strength was undeveloped. The general scheme outlined has been used in our laboratory for ten years. When we began work on that method, our thought was to give a particular type of color the number of rubs which would be comparable to average economical factory practice. Probably not ten per cent of the color consumed is finely ground. Of course when colors are used in enamels they are ground fine, but I am speaking of the total color production. The result is that in determining the number of rubs we tried to make the degree of development comparable to the degree of development that the average plant would give it if it were running on a fairly economical basis.

MR. NELSON.—In other words, it is entirely outside the difference you might get due to differences in the dispersion of the zinc oxide?

MR. BROWN.—We try to take care of that so that there is no variable by grinding on the roller mill, as we feel that we then have 100 per cent dispersion. We probably do not get that, but the variable on the other is so much greater that any difference would not have a practical significance.

MR. STAUDERMAN.—I should like to ask one more question; conclusion No. 9 of the paper refers to tests that should be dropped.

MR. LOVE.—The reason for this recommendation is that the bronzing test is special and not general, and is dependent on the type of vehicle, type of pigment and other variables. It was felt that only general specifications should be included. It would be better to allow special tests of this type to be arranged between manufacturers and consumers of the pigment.

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[1] Chief, Paint and Ceramic Research Division, New Jersey Zinc Co., Palmerton, Pa.

MR. BROWN.—Usually this bronzing and floating is a vehicle problem, not a pigment problem, and I think that it complicates the situation to consider it a pigment problem. Every day we see the same batch of some color that will float in one vehicle and will not float in another.

MR. STAUDERMAN.—But take, for instance, the manufacturer who is buying his material and has set up his standard of a special blue; this blue performs rather well in a given liquid, and a shipment this month might perform just as well as standard and next month you might notice a remarkable tendency toward bronzing.

MR. BROWN.—That tendency, however, is not apparent in the wet paint. When I think of bronzing on a blue, I have in mind the metallic sheen that develops on weathering. We no longer make any blue comparisons in the paste form. Formerly we did so but found that the paste might look all right, but not the enamels made therefrom. In an enamel made of a very dark color, such as blue, we can see differences that are not apparent in paste comparisons. The bronzing will not be apparent in the paste, as the eye does not register it on a small expanse. But by painting out a considerable expanse of an enamel and paint, we can see differences that we would never see in the paste.

MR. BONNEY.—According to Mr. Stutz, with a white pigment of a certain type, variations in consistency make relatively little difference in tinting strength. On the other hand, Mr. Ayers' paper shows that minor differences in procedure, in the vehicle used, in the amount of rubbing, make very large differences in tinting strength with the colored pigments. We may have perhaps six or seven white pigments as against 30, 40 or even 50 colored pigments to be tested for tinting strength. There is a real weakness in our testing methods for tinting strength of colored pigments. Mr. Ayers' paper indicates that some 50 rubs on a muller develop as much or more tinting strength than will be obtained by the greatest amount of milling obtained in a plant by ordinary rolls. But in the laboratory we take a specimen and mull it 150 times. One pigment in 15 or 20 mulls may develop the full tinting strength as much as will be secured in the mill. Another pigment may require the full 150 mulls to develop the tinting strength. The two pigments look the same on a laboratory test with 150 mulls, yet when put through the plant mill, one may develop half the strength of the other.

In addition to the number of mulls, we have the question of the vehicle; we have carbon paper, printing ink, paint and only one test for tinting strength for color of pigment, and that is 150 rubs of the muller. I feel that we are decidedly weak on that point, since all the evidence indicates that 150 rubs of the muller do not give us a practical relationship between the two pigments as they are used in our various processes, and I feel that we need additional work on testing methods for strength of colored pigment.

MR. LOVE.—Our plans for the future include the determination of the various factors which Mr. Bonney has discussed, a few of which have already been referred to in Mr. Ayers' paper.

[See also Discussion on Tinting Strength of White Pigments, page 531.—ED.]

TINTING STRENGTH OF WHITE PIGMENTS

BY R. H. SAWYER[1]

SYNOPSIS

This paper discusses the meaning and practical worth of tinting strength values for white pigments. It is shown that tinting strength does not afford a practical scale for the comparison of pigment types but does have more definite use in the routine comparison of similar pigments within a single pigment type. However, even with this limitation the tinting strength value frequently fails to be a good measure of hiding power. The best justification for the use of a tinting strength test as a measure of white pigment quality or uniformity appears to be the ease and speed of carrying out the test as a routine. The use of a simple test and the avoidance of certain unnecessary complications are favored.

The tinting strength of a white pigment is regarded as an important and significant property. Some determination of tinting strength is included in the evaluation of the great majority of pigment samples tested in the control and research laboratories of pigment manufacturers and users. A tinting strength test is a convenient test to use for judging the uniformity of production or shipment or for the evaluation of new pigment samples prepared in the course of pigment development research or offered to the pigment consumer.

Several methods have been devised for the determination of the property described as tinting strength. Gardner(**1**)[2] lists no fewer than nine. These range from the extremely simple, wholly practical to the cumbersome "exact." The problem of the selection of a suitable standard method has received the attention of the Society. Coöperative testing work carried out several years ago resulted in the adoption of the A.S.T.M. Tentative Method of Test for Tinting Strength of White Pigments (D 332 – 31 T).[3] A modification of this method is again being proposed.[4]

The problem of the development or selection of a suitable method for the tinting strength determination is by no means simple. This problem has at least two important aspects: The practical one of determining the meaning of the results and the use to which they are to be put, and the theoretical or fundamental one of precision. In the search for precision the more important first aspect seems to be ignored. An analysis of the practical meaning and use of the test is desirable.

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[1] Chemist, Krebs Pigment and Color Corp., Newport, Del.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 520.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 797 (1931); also 1933 Book of A.S.T.M. Tentative Standards, p. 525.

[4] See Report of Committee D-1 on Preservative Coatings for Structural Materials, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, pp. 400, 407 (1934).

The tinting strength of a white pigment is of considerable practical importance because it is conveniently determined and presumably affords an index of quality or uniformity. The practical use of tinting strength is, of course, predicated on the assumption that a higher tinting strength means a better or more valuable pigment. We may well ask, however, is a pigment of higher tinting strength a better pigment?

The tinting strength is determined presumably by the light reflecting power of the pigment and is therefore allied to other optical properties of the pigment likewise dependent on light-reflecting power such as hiding power, brightening power (as defined by Haslam(3)) resistance to slight contamination, and resistance to deep tinting. It is because tinting strength is closely allied with hiding power and has been assumed to be a measure of the latter that the use of tinting strength as a quality index has been justified. In fact, the present tentative method of the Society is supposed to yield results which can be calculated directly in terms of hiding power.

There has been a considerable difference of opinion as to whether tinting strength is or can be a measure of hiding power. Hallett(2) has held that there is a direct and quantitative relation, while Gardner(1), and Haslam(4) have maintained differently. It appears reasonable to believe that an exact parallelism cannot exist for practical purposes. One very important reason for this lies in the relation of each to brightness whereby tinting strength and hiding power change oppositely as brightness is changed. In addition, the initial brightness of a pigment need bear no direct relation to the brightness of the mixture in which it may ultimately be used, while this latter will have a very significant effect on hiding power. There are, however, very likely other fundamental reasons for a lack of a true direct relation. One such is very probably connected with particle size relations. The tinting strength test at present invariably uses a tinting pigment of finite particle size. This may easily result in the requirement that the optimum particle size to hide the tint to the maximum extent may be significantly different from that required to give best results in hiding a surface. Such an effect as this might be overcome by the use of a liquid tint of zero effective particle size. Other fundamental differences might also be cited. Still others may exist unrecognized.

Apart from the fundamental reasons for the lack of a relation between tinting strength and hiding power there is the very practical consideration of the use of the pigment. What, for example, is the hiding power of titanium dioxide? Hallett(2) showed that the hiding power of titanium dioxide can be calculated from its tinting strength, measured in a specified way. He gave 115 sq. ft. per lb. as the hiding power. It has been shown, however, that hiding power is very definitely a function of conditions of measurement and particularly of concentration of the pigment in the film. We have found(6) that the hiding power of titanium dioxide can be varied 50 to 100 per cent within the practical limits of enamel or lacquer formulation. Jacobsen and Reynolds(5) have found the same general effect. The hiding power of titanium dioxide or of any other pigment is, within quite broad limits, what you wish to make it or, rather, is determined by the kind of paint in which it is used and the hiding power developed by the paint. These factors determine the concentration of the pigment which must be used. The concentration, in turn, determines the unit hiding power of the pigment—its efficiency.

However, this is somewhat beside the point. We recognize that gross differences in tinting strength are indicative, qualitatively, of similar differences in potential pigment hiding power. These gross differences cannot be considered as quantitative and are, in fact, rarely so used. Tinting strength as such does not provide a suitable scale for the comparison of pigments of different type. It matters little whether titanium dioxide has 6.0 or 14.5 times the tinting strength of white lead—to quote extreme results in the cooperative work of 1929 to 1930. The fact that titanium dioxide is 6.0 to 14.5 times better than white lead in tinting strength might persuade a paint man to try it but his decision to use one pigment in place of the other would be based on a trial in which tinting strength would play no part and hiding power would be subordinated to other general paint properties. This is true even for pigments as close together as the high-strength lithopones and titanium-calcium pigment which differ by some 10 per cent in tinting strength.

The most frequent use of tinting strength, however, is not for the estimation of gross differences between pigment types but is usually for the estimation of small differences within a single pigment class. It is here that the test serves as a handy tool. How accurately does tinting strength afford a prediction of pigment value in use? Does it detect non-uniformity with certainty, reject only the bad, and approve only the good? Here again we expect the test to predict hiding power value primarily. In this case the fundamental reasons for discrepancy still apply. Concentration effects in relation to hiding power do not apply particularly in this case.

The work falls into three categories: Control of production, control of shipments, and guidance of research. For the control of production there does appear to be a useful relation between tinting strength and hiding power. The author has analyzed the results of tests on somewhat more than 100 samples representing three pigment types (each type considered separately) for which we have tinting strength and hiding power data. Tinting strength was determined by our standard laboratory method which very closely resembles the optional procedure in the present proposed tentative method. Hiding power was determined by a dry film method developed in the laboratory of the Krebs Pigment and Color Corp.(7). The samples were considered in four groups of twenty or more samples in each group. For each group a linear relation between strength and hiding power was calculated. The deviation of the individual samples from this linear relation was computed in terms of hiding power. The following table shows the frequency distribution of these devitations:

Deviation, per cent	Frequency	Deviation, per cent	Frequency
0	8	6	7
1	19	7	5
2	21	8	3
3	13	9	3
4	15	10	1
5	11	11	1

Total observations = 107.
Average deviation = ± 3.4 per cent.

From these data we see that tinting strength does, on the average, agree very well with hiding power. Consequently the average tinting strength of several samples might well serve for production control purposes in indicating uniformity or possible drift in quality resulting from a known process. However, a drift in hiding power could take place and still escape detection. This will appear more clearly in the following.

The control of shipments received or the guidance of research constitute a different type of problem in that we are most frequently concerned with single samples. In addition we may have no knowledge of the history of samples or may know that they represent significant departures from normal operation. Inspection of the data above shows that individual samples may show deviations from the normal relation of as much as 10 per cent. In fact, the original data show up to 18 per cent difference in hiding power at the same tinting strength, while differences of 10 to 15 per cent are not uncommon.

We had occasion at one time to carry out an extended comparison of samples of pigment type prepared by two radically different methods. In this case hiding power was measured by a dry film method and also by the cryptometer. The pigments made by either method showed a nice relation between tinting strength and hiding power but hiding power for the one method averaged about 15 per cent lower than for the other at equal tinting strength.

Hiding power was measured by the cryptometer for a series of laboratory pigments prepared to study the effect of a single variable in the preparation of the pigment. A linear relation between strength and hiding power was found. However, four commercially prepared pigments of the same chemical composition and apparently similar pigment properties showed hiding powers 25 per cent lower than the tinting strength – hiding power relation developed for the laboratory preparations would have indicated.

We are therefore forced to conclude that tinting strength does not afford a practical means for estimating with any certainty the quality in terms of hiding power of single pigment samples either in terms of a relation to supposedly similar samples or relation to a general type. The use of a tinting strength test in general is somewhat unsafe. Other practical tests should augment or supplant tinting strength.

Despite the general insecurity of tinting strength results, the convenience of tinting strength tests will undoubtedly cause the rather general use of tinting strength to continue. What, then, constitutes the best type of test? Two factors at once come to the fore, convenience and reproducibility. The ideal test should be easy and rapid, should not give unnecessarily misleading results, of course, and should be capable of giving reproducible results. No more in the sense of precision or exactness need be asked. Let it be recognized that from a practical standpoint an "exact" tinting strength and a comparison of pigment types in terms of tinting strength are meaningless.

If these points are recognized, the measurement of tinting strength can be relieved of two features which have complicated the tentative method unnecessarily. These are the requirement of constant light reflection from the paste and equal paste consistency. For routine gradings it is much more rapid to use a standard series of pastes against which individual sample pastes are graded. Within a single pigment type this approximates constant light reflection more closely than tinting strength, measured either way, approximates hiding power. A method employing equal paste consistency introduces the additional uncertainty of the operator's judgment, thereby reducing reproducibility and at the same time may distort the results in such a way as to decrease the chance of a correlation with practical value. On the other hand, the use of a constant ratio of pigment to oil is favorable to reproducibility and better conforms to pigment use.

The choice of the tinting pigment is, perhaps, less important. However, it has been our experience that the use of ultramarine blue tends toward much better reproducibility in grading, decreases the disturbing effects of tint differences, and renders results less affected by differences in the wetting characteristics of the oil used.

A clearer understanding of the problem of determining the value of a pigment in terms of its optical properties, will, I believe, lead to the conclusion that tinting strength does not afford a safe and satisfactory measurement. If, however, it is still desired to use tinting strength for control purposes, it should be done with caution. If tinting strength must be used, a simple test best serves the purpose and should be specified. Something similar to the optional procedure of the present proposed tentative method is adequate and satisfactory. The complications of the present tentative method should be eliminated and a practical method set up which will be fully as useful and certain as any method yet devised.

REFERENCES

(1) H. A. Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Sixth Edition, pp. 60–72 (1933).

(2) R. L. Hallett, "Hiding Power and Tinting Strength of White Pigments," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 895 (1930).

(3) G. S. Haslam, "Evaluation of Contrast-Obliterating and Brightening Power of White Pigments," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 2, p. 69 (1930).

(4) G. S. Haslam, Discussion on Hiding Power of Paints, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 903 (1930).

(5) A. E. Jacobsen and C. E. Reynolds, "A Practical Method for Determining the Hiding Power of Paints," Paper presented before Eighty-seventh Meeting, Am. Chemical Soc., Spring, 1934.

(6) R. H. Sawyer, "Comparison of Wet and Dry Film Hiding Power Tests," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 6, p. 113 (1934).

(7) R. H. Sawyer, "A Method for Measuring the Hiding Power of Dry Paint Films," Paper presented before Eighty-sixth Meeting, Am. Chemical Soc., Fall, 1933.

[For Discussion on Tinting Strength of White Pigments, see page 531.—ED.]

TINTING STRENGTH OF WHITE PIGMENTS

By G. F. A. Stutz[1]

Synopsis

It is suggested that tinting strength of white pigments can be exactly defined only in terms of the method of test used in determining such strength. It is further suggested that the method is of value chiefly in determining the uniformity of successive shipments of material and should, therefore, be as simple and rapid as possible with accuracy. A study of the factors influencing the test includes a consideration of the incorporation of the pigment in the oil, the consistency of the pigment-oil mix, the magnitude of the tint, method of viewing, and the strength computation. The conclusions indicate that the optional procedure of the new proposed Tentative Method of Test for Tinting Strength of White Pigments is the most desirable method of test.

Introduction

The Society has defined tinting strength[2] as "the power of coloring a given quantity of paint or pigment selected as a medium standard for estimating such power." The use of the term "coloring" would give the impression that tinting strength is a property associated only with colored or tinted pigments but the definition may be, and has been, construed in broader terms to include white pigments. The conditions existing in such a mixture of white pigments have been considered by Merwin[3] and further by Briggs.[4] The real significance of this property with respect to white pigments has been the subject for considerable debate. Thompson[5] has endeavored to rationalize the subject and to develop an equation representing the relationship existing between brightness and amount of tinting material. A relationship is found but it is entirely an empirical one relating only to a pair of white pigments. Judd[6] in the discussion of Thompson's paper has further discussed the factors involved and concludes with the observation regarding the Thompson relationship that "it refers only to the precise method used for the mixing of the pigments." It is evident that the results obtained in any determination of tinting strength are dependent upon the method used. The effect of variations has been recognized and Brown[1] has discussed in detail some of the precautions necessary in carrying out the manipulation. Calbeck and Olander[2] have attempted to overcome variations, particularly those due to initial brightness of the pigment by the use of only a small amount of tinting material, measuring the brightness on the Pfund colorimeter.

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[1] Chief, Pigment Research Division, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

[2] Standard Definitions Relating to Paint Specifications (D 16 – 24), 1933 Book of A.S.T.M. Standards, Part II, p. 735.

[3] H. E. Merwin, "Optical Properties and Theory of Color of Pigments and Paints," *Proceedings*, Am. Soc. Testing Mats., Vol. XVII, Part II, p. 496 (1917).

[4] T. R. Briggs, "The Tinting Strength of Pigments," *Journal of Physical Chemistry*, Vol. 22, p. 216 (1918).

[5] G. W. Thompson, "The True Tinting Strength of White Pigments," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 924 (1929).

[6] D. B. Judd, Discussion on Tinting Strength of Pigments, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 931 (1929).

In view of the recognized effect of variations in procedure, it would seem to be most exact to define tinting strength, at least in the case of white pigments, in terms of the method of test used rather than in any general term such as given by the A.S.T.M. definition.

With respect to white pigments, the property of tinting strength, as such, is only infrequently of interest. Occasionally manufacturers of cold water paints or of some special products require a measure of actual tinting strength of the white pigments which they use. More often the property is used only as a means of judging the uniformity with respect to optical properties of successive shipments of pigment. No importance can be attached to the determination from the standpoint of using it to judge the performance characteristics of a white pigment when that pigment is incorporated in paint. Moreover, most of the discrepancies to be found in tinting strength tests are due to an attempt to compare two different types of pigments, a comparison which is of questionable significance.

In this paper, tinting strength is considered from the standpoint of its being a routine test used to judge the uniformity of successive shipments of one single type of pigment. It has been successfully used by both pigment manufacturers and pigment consumers for this purpose for a number of years. An attempt will be made to outline some of the factors causing inaccuracies in the method and to indicate the most satisfactory procedure to be used from the standpoint of both speed and accuracy. For the purpose of routine testing, a simple, accurate, and rapid method is required.

EXPERIMENTAL PART

Incorporation:

Probably the greatest single factor influencing the determination is the method of incorporating the pigment in the vehicle. It is common practice to use a sample of 2 or 3 g. of white pigment and to incorporate this on a rubbing surface consisting of a lithographer's stone or a ground plate glass. Some operators use a flexible spatula with a 6-in. blade for the incorporation of the pigment in oil; others prefer a standard type glass muller having a grinding face of approximately 3 in. in diameter. In some cases, the muller is weighted so that the pressure applied by the operator may be constant and uniform. Experience in the laboratory of The New Jersey Zinc Co. indicates that the 6-in. spatula will give variable results unless used by an experienced operator. The results obtained by an operator using the spatula are of questionable value until the man has had at least three months' experience.

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[1] A. F. Brown, "Pigment Colors and Their Use in Protective Coatings," *Paint, Oil and Chemical Review*, March 5, 1924, p. 10, and March 12, 1924, p. 10.

[2] J. H. Calbeek and C. P. Olander, "An Application of the Pfund Colorimeter to the Determination of Tinting Strength of White Pigments," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 515 (1922).

To determine definitely the effect, and relative value, of the several methods, five operators were asked to carry out the determination on a sample of lithopone. The results obtained in this test are shown below:

Method of Rubbing	Operator No. 1, per cent	Operator No. 2, per cent	Operator No. 3, per cent	Operator No. 4, per cent	Operator No. 5, per cent
Steel spatula, 5 min	100	100	105	102	105
Regular muller, 100 rubs	101	100	101	101	102
15-lb. weighted muller, 100 rubs	102	100	101	101	101

In each case, a standard and the unknown are rubbed. Operator No. 1 is a man of long experience with all three methods and can check himself regularly within 1 per cent; No. 2 is also experienced in the determination but rubs samples only infrequently; No. 3 is a laboratory technician, having no previous experience with the test; No. 4 is a college graduate and paint chemist, having no previous experience with the test and No. 5 is a physical chemist and Ph.D., having no previous experience with the test. Accepting operator No. 1's results as correct, it is evident that the muller is superior to the spatula for an inexperienced man. The weighted muller offers no advantage over the regular muller.

The results shown indicate that the lithopone used has a greater possible strength than is indicated by the normal amount of rubbing given the sample by operator No. 1. Other samples of lithopone and other pigments may have an even greater potential strength not indicated by normal rubbing. Extreme rubbing with the steel spatula or prolonged rubbing for a period of as much as 40 to 60 min. with the muller will bring out this potential strength in the pigment. It may be argued that the ultimate strength is the only property which can be definitely determined since any intermediate amount of rubbing cannot be exactly defined.

However, this property of developing partial strength with normal rubbing and ultimate strength only with extreme rubbing is a characteristic of all pigments of the precipitated type, due to their aggregated structure. The degree of such aggregation may vary with different pigments, but it cannot be expected that it will be completely broken down with normal paint mill grinding. It would therefore seem to be more desirable to give the pigment a limited amount of mulling in an effort to approximate the incorporation resulting from the average paint mill grind.

Table I.—Tinting Strength Determinations.

Method of Dispersion	Relative Tinting Strength (Referred to 100 Rubs With Unweighted Muller), per cent
Zinc Oxide	
100 rubs, unweighted muller	100
5-min. rubbing, 15-lb. weighted muller	100
Rubbing to peak, 15-lb. weighted muller (required 10 min.)	102 to 103
Laboratory mill, Buhrstone type (close setting)	102 to 103
Laboratory roller mill (two passes)	102 to 103
Basic Carbonate of White Lead	
100 rubs, unweighted muller	100
5-min. rubbing, 15-lb. weighted muller	106 to 107
Rubbing to peak, 15-lb. weighted muller (required 25 min.)	111 to 112
Laboratory mill, Buhrstone type (close setting)	93 to 94
Laboratory roller mill (two passes)	93 to 94

To determine the relationship between degree of mulling and paint mill incorporation, two pigments were ground in oil by five methods of dispersion and the relative tinting strength of the resulting paste determined. The results are given in Table I.

Zinc oxide is an example of a pigment of minimum aggregation and of easiest dispersion. One hundred rubs with the unweighted muller are seen to be practically equal to maximum paint mill grinding. Basic carbonate of white lead, on the other hand, is an example of an extremely aggregated type of pigment and 100 rubs with the unweighted muller are slightly superior to paint mill incorporation. Considering these two pigments as extremes, it would seem reasonable to use 100 rubs with the unweighted muller as an average for white pigments.

Since the amount of rubbing or mulling is a definite factor in determining strength, particularly with aggregated pigments, it follows that any set of conditions permitting the operator to do more work on the pigment will show a corresponding advantage for that pigment. If the pigment is rubbed in the form of a relatively heavy or thick paste, more work will be accomplished with 100 rubs of the muller than if the pigment were rubbed as a thin paste. A comparison of three samples of zinc sulfide of differing oil absorption rubbed with the same amount of oil was made to determine the magnitude of this effect. The results are shown below:

	Oil Absorption, ml. per 20 g. (Gardner-Coleman)	Relative Tinting Strength, per cent	
		5-min. Rubbing	Rubbing to Peak
Zinc sulfide No. 1	5.3	100	105
Zinc sulfide No. 2	4.1	93	105
Zinc sulfide No. 3	4.2	94	105

All samples, by prolonged rubbing, attained the same maximum of 105 per cent. However, the highest oil absorption, forming the stiffest paste, showed the greatest strength after only 5 min. rubbing, indicating that more work of dispersion could be accomplished in the heavier paste.

To determine further this effect of consistency of the paste on the development of strength, a sample of zinc sulfide, rubbed in each case for 5 min. with the weighted muller, gives the results indicated below:

Drops of Oil Added to Dry Pigment	Drops of Oil Added to Paste After Mulling	Relative Tinting Strength, per cent
45	0	97
40	5	99
35	10	100
30	15	101

In each case the same total amount of oil is present in the final paste but the amount of oil present during rubbing is decreased. With the heaviest consistency paste an advantage of 4 per cent in relative tinting strength is indicated. It would therefore appear to be most desirable to rub all pigments at the same consistency in order to perform equal amounts of work of dispersion with a definite rubbing procedure.

Since pigment dispersion is of major importance, the vehicle used may be expected to affect the result. The value obtained will therefore be a function of the vehicle used. This variable has not been studied in this paper. A bleached linseed oil of acid number approximately 5 has satisfactory dispersing power for the average white pigment.

Consistency:

It is well recognized that the amount of oil used in making the determination or the consistency of the paste obtained has a definite bearing on the result. The addition of one drop of oil to the paste may make a difference of several per cent in tinting strength value; the thinner the consistency, the higher is the relative tinting strength. It therefore appears to be most desirable to determine tinting strength at constant or equal consistency. However, from the standpoint of the routine operator, carrying out the determination at equal consistencies is laborious and time-consuming. The paste when prepared must be judged by some consistency test in comparison with the standard and if it does not equal the standard, additional amounts of oil or of dry pigment must be incorporated until equal consistency is obtained. For exactness, the pigment should be rubbed again with the amount of oil found to be necessary. The consistency may be specified as the point at which the paste can just be thrown from the spatula by a gentle whipping motion of the hand or by tapping the spatula blade with the index finger.

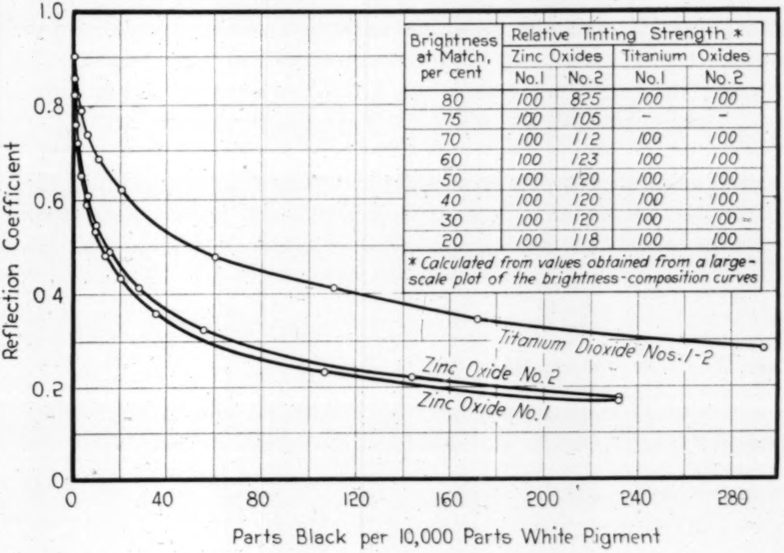
A much faster and simpler method of operation is to work at a constant pigment-vehicle ratio and take whatever variations in consistency may be obtained. To determine the magnitude of the error to be expected from the use of a constant pigment-vehicle ratio, two lithopones were selected representing the extremes in consistency characteristics of commercial material. Using the method of constant pigment-vehicle ratio, the thin consistency lithopone had a rating of 101 per cent of the standard. The heavy consistency lithopone had a rating of 102 per cent of the standard. When the consistency of the heavy lithopone was reduced to equal that of the thin lithopone, by the use of additional oil, the heavy material had a tinting strength of 107 per cent or an increase of 5 per cent.

In another case, two zinc oxides were selected representing the extremes in consistency characteristics for this material. Using the method of constant pigment-vehicle ratio, the thin consistency zinc oxide had a rating of 94 per cent of the standard. The heavy consistency zinc oxide had a rating of 106 per cent of the standard. By adding sufficient oil to the heavy consistency material to equal the consistency of the thin material, the rating was 113 per cent or an increase of 7 per cent.

While this effect of consistency on rating may cause some differences in absolute values, it would not appear to be of great importance in the routine grading of material since successive shipments of the same lithopone, zinc oxide or other pigments do not vary in consistency over the range shown above. Because of the greater speed and simplicity it would therefore appear most desirable and within a reasonable limit of error to use the method of constant pigment-vehicle ratio rather than constant consistency.

Tinting Material:

Some operators prefer to use the tinting material in the form of a paste to be incorporated with the white pigment after it in turn has been ground into a paste. Whether or not the use of a paste of the tinting material is necessary is dependent upon the type of tinting material used. When using a dry tinting material and rubbing it with the white pigment, the tinting material must be readily dispersed, and made to attain *its* maximum strength characteristics with the normal amount of rubbing given. A comparison was made of the dry addition and the paste addition using ultramarine blue, a tinting material of good dispersion characteristics. The results were identical.



Brightness at Match, per cent	Relative Tinting Strength *			
	Zinc Oxides		Titanium Oxides	
	No. 1	No. 2	No. 1	No. 2
80	100	825	100	100
75	100	105	–	–
70	100	112	100	100
60	100	123	100	100
50	100	120	100	100
40	100	120	100	100
30	100	120	100	100
20	100	118	100	100

* *Calculated from values obtained from a large-scale plot of the brightness-composition curves*

FIG. 1.—Brightness of Several White Pigments to Which Varying Amounts of Black Tinting Material Are Added.

It should be recognized that the use of different tinting materials may give somewhat different results. As an example, two samples of titanium dioxide used in this work were found to have identical strength, using black as the tinting material, but to have a difference of 10 per cent using ultramarine blue as the tinting material. Again the results can be defined only in terms of the method of test used and therefore the tinting material should be definitely specified.

Magnitude of the Tint:

The magnitude of the tint obtained or the reduction in brightness obtained has been considered an important factor and it is proposed to conduct all determinations at the same brightness of tint with a reflection value of 20 per cent. It is found that with different samples of the same type of material this factor is of little importance, provided the brightness is within the range of from 20 to 60 per cent in reflection coefficient. The results obtained in determining the brightness of several white pigments to which varying amounts of black tinting material were added are shown diagrammatically in Fig. 1. Two samples of titanium dioxide from different manufacturers were determined. Appreciable differences are found at high brightness, above 80 per cent. Below 60 per cent brightness the curves are identical. Two zinc oxides representing the extreme variation to be found from the standpoint of both strength and method of manufacture were determined. Again at high brightness the difference is great, but below 60 per cent brightness the relative strength is constant. It should be observed that these curves for zinc oxide actually intersect or cross one another at about 75 per cent brightness.

TABLE II.—RELATIVE ACCURACY OF TWO METHODS OF VIEWING.

	RELATIVE TINTING STRENGTH, PER CENT	
	VIEWED THROUGH GLASS	VIEWED AT SURFACE
Lithopone No. 1	100	100
Lithopone No. 2	100	100
Lithopone No. 3	110	108
Lithopone No. 4	97	96
Lithopone No. 5	98	98
High zinc sulfide lithopone No. 1	98	99
High zinc sulfide lithopone No. 2	97	96
High zinc sulfide lithopone No. 3	100	100
Zinc sulfide No. 1	100	99
Zinc oxide No. 1	105	105
Zinc oxide No. 2	110	108
Zinc oxide No. 3	102	101
Zinc oxide No. 4	106	105

Similar curves have been shown by Bruce, Calbeck and others comparing various pigments, and the indications are that the strength characteristics determined at different brightnesses would be somewhat different. It should be emphasized that this is true for a comparison of different pigments; for two pigments of the same type it appears to be of little importance.

Method of Viewing:

It is common to place the pastes on either a piece of clear colorless glass or bright tin. When judging color, it is recognized that viewing must be from the top or air interface. In the case of the tinting strength determination of colored pigments, it is generally considered that top viewing is necessary. However, when viewed through glass, smaller differences are readily perceptible and it is therefore easier for the operator to carry out the determination. An accuracy of from 1 to 2 per cent may be expected when viewing through glass; from 2 to 4 per cent when viewing from the air interface. To determine the effect of this factor, a number of samples were viewed both through the glass and from the top surface. The results are recorded in Table II.

It should be kept in mind that the strength determination when viewing at the surface is somewhat inaccurate. On this basis it would appear reasonable to conclude that, in the case of white pigments, viewing through the glass gives the same order of results as when viewing at the surface, and because of the greater ease and accuracy, this method is to be preferred.

Computation of Tinting Strength:

Because of the variation normally encountered due to differences in the brightness at which the determination is made, it has been considered desirable to reduce the unknown sample in each case to a brightness common with that obtained with the standard. Tinting strength is then computed as the ratio of the amount of tinting material added to the unknown sample to the amount of tinting material added to the standard times 100. This method requires the addition of varying amounts of tinting material to the unknown sample until its brightness is adjusted to equal that of the standard with a fixed amount of tinting material. If only one sample is being determined, this method is as satisfactory as any. However, if several samples of unknown material are to be determined, the reverse procedure is more desirable from the standpoint of amount of work required. In this case, varying amounts of tinting material are added to the standard sample to give a series of standards. A fixed amount of tinting material is added to each of the unknown samples and each is compared to the most nearly corresponding standard tint. Tinting strength is then computed as the ratio of the amount of tinting material in the unknown sample to the amount of tinting material in the standard sample times 100. In view of the constant brightness – tinting material relationship, indicated for any one type of material in Fig. 1, the results obtained by either method of computation should be identical. Whatever variations are present are reasonably small and it would seem desirable to accept these in favor of the greater ease and less amount of work involved in the second procedure.

CONCLUSIONS

Considering only a comparison of two pigments of the same type and method of manufacture and using the test only as a routine method of judging the uniformity of successive shipments of material, it is indicated that the following factors should be recognized in carrying out the method:

1. Rubbing should be done with an unweighted muller, 100 rubs being used (2-g. sample).
2. A constant pigment-vehicle ratio should be used. This ratio should be a definite quantity for each type of pigment.
3. The coefficient of reflection of the tint should be between 20 and 60 per cent. Some advantage in accuracy may be expected at the lower reflection coefficient.
4. Viewing should be through clear colorless glass panels.
5. Strength should be computed on the basis of variable amounts of tinting material added to the standard pigment.

Acknowledgment.—The author wishes to acknowledge the assistance of Messrs. D. L. Gamble and C. W. Farber in preparing the data and discussion.

DISCUSSION ON TINTING STRENGTH OF WHITE PIGMENTS

Mr. R. H. Sawyer.[1]—On page 523 of Mr. Stutz's paper, he refers to tests with the spatula, muller, and weighted muller on lithopone. Lithopone represents a pigment already well dispersed in a medium of lower refractive index. If a prime pigment such as zinc sulfide or titanium dioxide were used, one would expect that differences in work done on the paste resulting in different degrees of dispersion of the prime pigment in the medium of lower refractive index would show up more effectively as differences in tinting strength. Thus, differences between operators found to be 5 per cent with the spatula in the case of lithopone might be expected to be quite a bit greater with the prime pigments. I should like to ask Mr. Stutz whether he has had this experience or whether in the absence of actual data he would not regard the work with lithopone as representing a favorable case.

Mr. G. F. A. Stutz.[2]—We have not carried out the comparison of the spatula, muller and weighted muller with a prime pigment as, for example, with zinc sulfide and therefore cannot answer Mr. Sawyer's question. If we may be permitted to express an opinion, we would expect that the differences would be even greater with a prime pigment such as zinc sulfide.

Mr. A. F. Brown.[3]—If I may draw on our color experience, I would say that the stronger the potential strength of pigment, the greater the error when a spatula is used. We can make a fairly accurate test on a reduced green with a spatula, but if we take the pure form of that same color, we will have a very large error. I like to consider the muller as an instrument which enables one to apply with greater accuracy, the amount of energy used in grinding the pigment on the slab—trying to duplicate in a small way what the user does in a large way. With strong pigments (if we consider lithopone as a weak pigment as compared with titanium dioxide), I would think that in a strength test with a spatula, the error would be greater.

Mr. Stutz referred to the question of consistency, and in this connection I am wondering if the white pigment industry might not use the methods we employ on some of our color pigments. In our routine work, the way we check consistency is to make an actual grind on a small roller mill. The small roller mill is probably one of the most useful pieces of equipment we have. Kent is now making a mill 3 in. in diameter and 5 in. long, and if we are trying to draw very fine distinctions between the relative oil absorption of two different pigments of the same type, we simply grind very stiff pastes using heat-bodied oil as the vehicle, and it is remarkable the minute differences one can detect in this way. On these small mills two or three grams of material can be ground very accurately; I am speaking of a color that has a higher oil absorption than most of the white pigments, but enamels can be made with 5 g. and a formula worked out that can be used in making a 500-gal. batch. One can change from one pigment to another with no contamination. The mill is easily cleaned and is a most satisfactory piece of apparatus.

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[1] Chemist, Krebs Pigment and Color Corp., Newport, Del.

[2] Chief, Pigment Research Division, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

[3] General Manager, Imperial Color Works, Glens Falls, N. Y.

MR. G. W. THOMPSON.[1]—May I make one comment on Mr. Stutz's paper. In referring to the definition of color there was a slight slur on the definition of tinting strength; where the word color is used in that definition, it meant either chromatic color or achromatic color. The Optical Society of America has divided all colors into two classes, achromatic and chromatic. The achromatic includes all whites, grays and blacks; the chromatic colors are those that possess hues.

On the general question of tinting strength, I do not believe that any paint manufacturer or manufacturer of white pigments has ever had a more useful test for indicating the value of a white pigment and its deficiencies and for studying and improving his product. I know that has been the case with the National Lead Co. Nearly 42 years ago that was one of the first tests I put into use, and it has been exceedingly valuable to us.

We may ask what the factors are that determine tinting strength. And we might as well ask, along with it, what the factors are that determine hiding power. One of the factors is refractive index. Another factor is degree of fineness. We can take a comparatively coarse pigment and mix it as in a tinting strength test with some black or some blue, and put it through a grinding operation and it becomes lighter or brighter with continued grinding, so that we know that fineness is one of the factors in determining tinting strength. We know that brightness is one of the factors that determines tinting strength. We know that the refractive index and fineness are elements in determining hiding power. We know, however, that there is an inverse relation of brightness in these two tests as applied to white pigments; the brighter the pigment, the higher its tinting power but the lower its hiding power. On the other hand, the lower the brightness of pigment, the greater the hiding power. Now that is apparently the only difference. I do not mean to say that we know all about tinting strength nor all about hiding power, but so far as I can see, that is the only element of difference.

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[1] Chief Chemist, National Lead Co., Brooklyn, N. Y.

We should like to know more of what the effect of dispersion is, both on hiding power and tinting strength. Sometime it will be determined just what that effect is, but it seems as though it ought to have the same effect on tinting strength as on hiding power. I cannot help but think that tinting strength as well as hiding power are methods of measuring the value of pigment. I know years ago Mr. F. G. Breyer impressed upon us that it is the physical characteristics of pigments that determine their value—not their chemical composition. Now certainly hiding power and tinting strength are physical characteristics just as specific gravity is. So too refractive index and fineness of division are physical characteristics. I do not think we have the final answer, but I cannot see why there should be any objection to the use of the tinting strength test in evaluing pigments.

MR. SAWYER.—Mr. Thompson points out that tinting strength and hiding power are alike in depending upon refractive index and fineness although not alike in relation to brightness. From this he infers that a measurement of tinting strength is equivalent to a measurement of hiding power. It is true that the two properties are alike in their gross relations to certain other properties of the pigment. However, we cannot escape the experimental evidence that this gross parallelism breaks down seriously in some cases. We do not know what factors to hold responsible but we have sound reasons for believing that hiding power and tinting strength as now measured are not necessarily parallel functions. Consequently the present tinting strength tests may lead to erroneous conclusions regarding the hiding power values of pigments.

Thus the paint manufacturer may be led by tinting strength to accept inferior pigments or to reject superior ones. The pigment manufacturer using tinting strength as a guide in his work toward pigment improvement may secure improved tinting strength by sacrificing hiding power.

MR. STUTZ.—As to the reason why this relationship is not exact and does not hold, it seems to me that the relative amount of pigment and of vehicle that is present in the two tests has some bearing. We know that a pigment becomes more efficient in both tinting strength and in hiding power as it is diluted down more.

MR. THOMPSON.—That of course represents another parallelism.

MR. STUTZ.—Mr. Steele, of our laboratory, working with paper where the pigment is diluted down a great deal more than it is in the case of paint, has found that he can come much closer to applying the recognized laws of optics to the opacity concentration relationship that he gets, and it seems to me that, as we go up in the concentration of pigment, as we do in the tinting strength test, we are going more and more into the range where recognized optical laws do not hold and where the pigment is least efficient. That may possibly explain the discrepancy.

MR. THOMPSON.—I should like to ask Mr. Sawyer, if I may, whether in making a tinting strength test, he adds, say, a small amount of black, does not the white hide the black in pretty much the same way that the white pigment would hide a black surface?

MR. SAWYER.—That of course is true. I am not denying a broad general relationship between the two, I am denying a useful practical specific relationship between the two, applying to individual samples or to different types of pigment, where pigments differ expectedly or unexpectedly in certain properties, many of which we do not measure. It is not for the pigment manufacturer to favor dropping the tinting strength test or to question it; the paint man should be the one to do that, because the tinting strength test may lead him into trouble. We can develop our pigment to improve tinting strength, but suppose we do develop it until we have secured considerably better tinting strength, is it not possible that in so doing we may have lost hiding power? Our pigment, after that development, is less valuable in use.

MR. A. E. JACOBSEN.[1]—Mr. Sawyer believes that it is unsafe to use tinting strength for estimating hiding power. In order to determine hiding power, some suitable method of testing hiding power must be employed. Otherwise it is impossible to say that the pigment showed a hiding power value 25 per cent lower than tinting strength in the tinting strength - hiding power relation. I believe there is less controversy in regard to tinting strength than there is with regard to hiding power. We have made a number of tests by different hiding power methods, and find that the hiding power value of pigments is dependent upon the pigment volume concentration. Tinting strength also depends on dilution. If that is the case, why is it not possible to have a tinting strength method, the results of which could be related to hiding power? I should like to ask Mr. Sawyer what he thinks of using the cryptometer as a method of test for use in the comparison of tinting strength with hiding power.

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[1] Chemist, Titanium Pigment Co., Inc., New York City.

MR. SAWYER.—As I understand it, the purpose of this discussion is to evolve ideas regarding testing methods in general which may direct possible future work along profitable channels. So far, we do not have a satisfactory hiding power method. However, if it is agreed that tinting strength is a poor method of testing for hiding power, that should serve to direct attention toward securing a better hiding power test. As to the relation between hiding power and tinting strength, it depends possibly on the method of test as to whether one gets a certain parallel relation.

Answering Mr. Jacobsen's question, I do not favor the use of the cryptometer as a method of test for hiding power. I favor the incomplete hiding method. In the particular instances I am discussing the two give essentially similar results.

I might add that in the cases which I cited, we were able to show a straight-line relation between tinting strength and hiding power for one group of pigments, and for another group of pigments. Taking two groups of lithopones that have been prepared differently, both gave straight-line relations by complete and incomplete hiding methods, yet the two straight lines were apart by approximately 15 per cent. The results were such as to indicate that uncertainty in the tests was not responsible for the difference observed.

MR. THOMPSON.—I should like to ask Mr. Sawyer a question; he said he feared that if he worked to increase the tinting strength, he might find that he had reduced the hiding power; has he ever come across a case of that kind where he had increased tinting strength and impaired the hiding power? We have found such a thing as fictitious hiding power or tinting strength. I do not want to go into the details of that, but in such a case you have done something else with the pigment besides increasing its tinting strength; you have increased its oil-taking power, in all probability, or its surface relations with the vehicle have been changed. I know of a pigment that will have 1500 tinting strength compared with white lead 100. Grind it and it goes down to 1000.

MR. SAWYER.—In the particular case I cited showing the difference of 15 per cent between the two straight-line relations, the relationship which gave high tinting strength and lower hiding power was an improved process, supposedly. In other words, using our lithopone scale, which puts lithopone at approximately 130 on the tinting strength scale, we could improve the tinting strength of lithopone to between 145 and 150, and yet find that it came out no better than equal in hiding power to the usual product. If we improved it to 140, it was poorer in hiding power. If we had poor control of the process and were not able to improve it beyond 140, we found in our tests that the hiding power actually was impaired by such work. In the case of lithopone there appears to be some indication that peak hiding power and peak tinting strength do not correspond in terms of the particle size or some other properties of the pigment, and consequently, as we approach the peak for tinting strength, we may move away from peak in hiding power. That is largely speculation; I do not have much evidence to support it.

I should like to raise one more point. It seems to be felt that in comparisons of white pigments, comparisons should be made at equal consistency. Mr. Stutz says in his paper that, "It therefore appears to be more desirable." He is merely repeating what may be the consensus of opinion in testing. I disagree—dealing, of course, with a single type of pigment. Slight differences in consistency will produce no difference in the use of the pigments, and in the case of paints prepared from pigment of slightly different consistency, the ultimate dry paint film laid down will be of the same composition, and will show no differences related to consistency differences of the initial pigment used. Large differences in consistency will be met by the paint man in making his paints by an adjustment of his paints with thinners, involving again no change in the ultimate dry film laid down. Consequently, I think that pigments within a given type, and speaking exclusively of white pigments, should be compared at equal pigment oil ratios as best paralleling use in practice.

MR. G. E. WALKER.[1]—Three things should be considered while working out a new method of test. One is its effect on competitive selling; another is control of material in the process of manufacture; and the third is that of research test methods in general. The Society is interested in testing methods from all three points of view. The discussion would seem to me to indicate that tinting strength measurement has not gone beyond the research stage, or certainly not beyond the product manufacture control stage, requiring further research and study before developing into any form of specification.

MR. STUTZ.—Is it not better to develop a test for measuring directly the property in which we are interested, rather than develop a secondary test for measuring indirectly that property, demanding a correlation factor? Specifically, are we not better off to develop the hiding power test?

MR. R. L. HALLETT.[2]—Mr. Stutz has raised the question as to why we should develop a method for tinting strength in an attempt to interpret hiding power when we could perhaps develop a direct method for hiding power. If we could develop a quick efficient control and research test for hiding power, it would be very useful, but all of the hiding power methods that have been proposed are difficult and complicated. Tinting strength determinations on the other hand are quick and accurate and are being performed in large numbers every day in the laboratories of many paint and pigment manufacturers. The determination of tinting strength has been and continues to be a very valuable test for white pigments and is one of the principal tests relied on to control manufacture and maintain uniformity by those who make the pigments and to test the quality of deliveries by those who use the pigments. The consumer of white pigments may use tinting strength determinations principally to check deliveries against a standard, but even if the test is used in this way the consumer has always had in mind that tinting strength indicates something of value in the pigment. The tinting strength of a white pigment, if it is only a measure of tinting strength, is of no particular importance as no consumer buys a white pigment because of the amount of coloring or tinting material required to give certain tints in a paint mixture, but he does realize that tinting strength measures some pigment characteristic of real value to him, and that valuable characteristic is hiding power. In general the laws of optics apply equally to tinting strength and hiding power. Refractive index is obviously a factor in both tinting strength and hiding power and it is equally apparent that specific surface or particle size affects both of them in the same way. Initial whiteness or brightness of a white pigment introduces a minor discrepancy because increased brightness increases tinting strength and decreases hiding power, but if we are considering reasonably high-grade white pigments with good competitive color, slight differences in brightness will become of theoretical rather than practical importance. Mr. Sawyer's paper discusses the changes in unit hiding power of a pigment caused by variations in proportions of pigment and oil in paint mixtures, but such variations have a similar effect on tinting strength. We might sum up by saying that the same laws of optics apply to both tinting strength and hiding power of the white pigments with the single exception of initial brightness, which is relatively unimportant in considering white pigments of good competitive color.

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[1] Technical Director, Southern Mineral Products Corp., Piney River, Va.

[2] Chemist, National Lead Co., Brooklyn, N. Y.

It seems evident that tinting strength is a good practical measure of hiding power, even if it will not always measure hiding power in exact fundamental units and I think most consumers of white pigments consciously or unconsciously consider this relationship to be true and value the tinting strength of white pigments for that reason. I have never known of a case where a white pigment with relatively high tinting strength did not have a correspondingly relatively high hiding power.

MR. STUTZ.—If I understand Mr. Hallett's remarks, the substance of them is that the experience of the National Lead Co. laboratories is that there is a definite relationship between tinting strength and hiding power, in spite of the experience of Mr. Sawyer that there is not. I should like to add that we can cite similar instances from our laboratory where the spread is 15 to 25 per cent. There may be something about the fact that these discrepancies have been found with lithopone. Our experience is that no parallelism can be drawn between the two tests, despite the possible inaccuracy in each test.

MR. THOMPSON.—If we will distinguish between indication and determination, there would not be so much difference between us. In England many years ago and perhaps now, in reporting an analysis they frequently gave an estimation, not a determination; there is quite a difference between estimation or indication and determination. Later on we may be able to determine, but that is a very different thing from indicating.

MR. H. A. NELSON.[1]—It seems to me that there is a difference of viewpoint that we must not forget. After all, we pigment manufacturers are interested in improving our products and want to endow them with the ultimate of useful qualities, so we, of course, have to look at these things very critically. The paint manufacturer may or may not accept our efforts at the value that we place on them, but after all some of us have found that we do not have a safe guide in tinting strength and feel that we must pay more specific attention to hiding power. It may be that the paint manufacturer does not need to pay attention to our work with hiding power now, but if he wants to take advantage of the developments the pigment manufacturer gives him, he certainly will have to include it in his testing schedule.

MR. A. E. STAUDERMAN.[2]—I believe that we should have the viewpoint of the paint manufacturer. It is up to him to determine, by the best means available, the value of each step taken in the testing. He may choose to examine shipments of pigments merely on a basis of mass tone or tint, to check for uniformity, or to note the oil absorption or fineness. For such work the method as outlined is ample. The question of hiding power is beyond the scope of the present method.

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[1] Chief, Paint and Ceramic Research Division, New Jersey Zinc Co., Palmerton, Pa.

[2] Director of Laboratories, Devoe & Raynolds Co., Brooklyn, N. Y.

In defense of the method, and more specifically of the "Optional Procedure," we avoid the term hiding power. I should like to emphasize that because the original committee discussed the point thoroughly when the work was first brought before the Federation of Paint and Varnish Production Clubs. We did not care to become involved in a discussion of hiding power in this specification. The method for developing mass tone and tint is quite simple and is based on the paint manufacturer's use of the pigments, which fortunately does not confuse tinting strength and hiding power.

We have various methods for determining hiding power of a pigment, and this is important especially when establishing a new source of supply. We utilize different methods and equipment and express results entirely independent of tinting strength.

MR. SAWYER (*author's closure by letter*).—I am quite pleased with this discussion. In it emphasis has been placed on the uncertainty of the relation existing between hiding power and tinting strength, because it is rather generally agreed that the chief reason for using tinting strength is that tinting strength is expected to be indicative of hiding power value. If the general user of tinting strength tests for white pigments will understand that the test may lead him astray in following quality, that ease and reproducibility are not enough, we then should have more attention given to performance tests in general and to hiding power tests specifically on the part of the pigment consumers. It is my hope that these papers and this discussion have been a step toward this understanding.

[See also Discussion on Tests for Mass Tone and Tinting Strength, page 509.—ED.]

MOTORS AND FUEL VAPOR PRESSURE

By T. A. Boyd[1]

Synopsis

The schedule of Reid vapor pressures for automobile gasolines proposed by the Section on Vapor Lock of Technical Committee A of the Society's Committee D-2 on Petroleum Products and Lubricants as being suitable for use at various maximum atmospheric temperatures is discussed, and exception is taken to it from four viewpoints, as follows: first, of the ambiguity of the term "maximum atmospheric temperature" which it contains; second, of the limitation of Reid vapor pressure as a measure of vapor locking tendency in that gasolines of the same Reid vapor pressure may give off different volumes of vapor at the temperatures reached by the fuel in service; third, of the belief that at least a portion of the schedule is out of line with current commercial practice; and, fourth, of the fact that, as data are presented to show, many cars now in service will give vapor lock on gasolines conforming to the proposed schedule. An alternative schedule is suggested. It is shown, also, that cars have been and are being improved in respect to freedom from vapor lock, and something of the magnitude of the obstacles which must be overcome in making such improvements is suggested.

Proposed Schedule of Reid Vapor Pressures:

It has been proposed by the Section on Vapor Lock of Technical Committee A on Gasoline of the Society's Committee D-2 on Petroleum Products and Lubricants that the Reid vapor pressure limits suitable for automobile gasolines be set thus:

For 100 F. maximum atmospheric temperature, 8 lb. at the filling station.
For 85 F. maximum atmospheric temperature, 10 lb. at the filling station.
For 75 F. maximum atmospheric temperature, 12 lb. at the filling station.

We have expressed the belief that the Reid vapor pressures given in this proposed schedule are too high and that this is particularly true in the lower portion of the temperature range within which it is thought that a greater degree of freedom from vapor lock should be provided for than at the higher temperatures. It is for this reason that we have been asked to prepare a discussion of the matter. Following, then, are given the principal reasons for the expressed belief, as just stated, together with data upon which the opinion rests.

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[1] Head, Fuel Section, Research Division, General Motors Corp., Detroit, Mich.

"Maximum Atmospheric Temperature" an Ambiguous Expression:

The first of the reasons for the belief that the Reid vapor pressures proposed in the above schedule are too high arises from the ambiguity of the term "maximum atmospheric temperature." It has been suggested that, because the temperatures given in the proposed schedule are *maximum* temperatures, they thereby automatically include a factor of safety which is thought to arise from the fact that only on rare occasions is the maximum temperature stated actually reached. But in reality that may not be the case, because there are two respects in which the expression "maximum atmospheric temperature" is capable of a variety of interpretations. There is, first, the question about whether it means the "absolute monthly maximum," the "average monthly maximum," or the "average daily maximum" of the weather bureau, or whether it means some other form of maximum taken over a different period. There is, second, the question about whether it means temperatures as recorded by the Weather

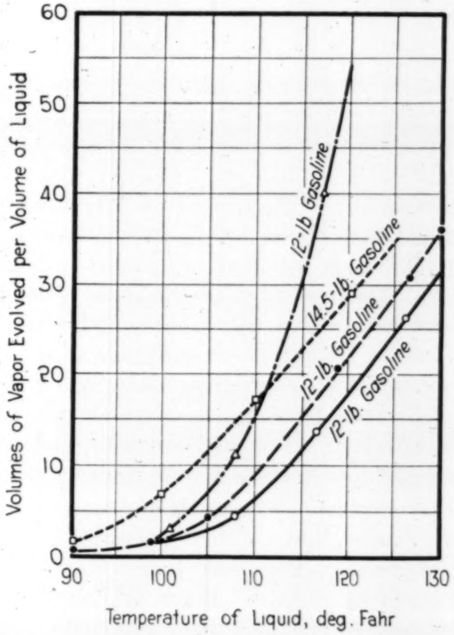

FIG. 1.—Volumes of Vapor Evolved at Different Temperatures by Three 12-lb. Gasolines and One 14.5-lb. Gasoline.

Chart showing that the Reid vapor pressure of a gasoline may not give a true measure of the amount of vapor which it will evolve at temperatures above 100 F.

Bureau or those actually existing where the car is driven, or perhaps where the filling station is located. This second question becomes one of importance because, in general, Weather Bureau temperatures are not so high as those which actually prevail on the roads and streets where cars are driven. Furthermore, regardless of what the intention of the schedule of safe limits may be, the inference of some interpreters of it will probably be that the temperatures named there are those at which the corresponding Reid vapor pressures are suitable for universal use, and not as temperatures which should not be exceeded.

Limitation of Reid Vapor Pressure as a Measure of Vapor Locking Tendency:

The second reason for believing that the proposed schedule is too liberal for the present is concerned with a fundamental limitation which Reid vapor pressure has as a measure of vapor locking tendency. The limitation referred to is that two gasolines which have the same Reid vapor pressure at 100 F. may evolve widely different volumes of vapor at the temperatures above 100 F. to which gasolines are often heated in the fuel-handling systems of cars (1).[1] The converse also is true, namely, that gasolines of different Reid vapor pressures may at a given temperature evolve the same volume of vapor. Thus, the data of Fig. 1 show that of three gasolines all of the same Reid vapor pressure, namely, 12 lb., one gave off by direct measurement in a displacement apparatus 18 volumes of vapor per unit volume of liquid gasoline at 120 F., a second 22 volumes, and a third over 50 volumes. In comparison with these figures, another gasoline of 14.5 lb. Reid vapor pressure evolved 29 volumes of vapor at 120 F. Bridgeman (2) has reported also that, of two gasolines which evolved the same amount of vapor at 120 F., one had a Reid vapor pressure of 11.1 lb. and the other of 13.8 lb. Since the cause of vapor lock is vapor, it is to be expected that the two gasolines reported by Bridgeman, although differing by 2.7 lb. in Reid vapor pressure at 100 F., would be substantially alike in respect to producing vapor lock at 120 F. And experience indicates that this is so.

It should be said, however, that this discussion of the limitation of Reid vapor pressure as a measure of the tendency of a gasoline to give vapor lock is presented here not because of any belief that Reid vapor pressure does not give a useful indication of vapor-locking tendency, but because it involves an unavoidable variable which constitutes an important reason for not setting the schedule of Reid vapor pressures too high.

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[1] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 549.

Relationship of the Proposed Schedule to Commercial Practice:

The third reason for taking exception to the proposed schedule is concerned with the fact that the Reid vapor pressures proposed for 85 F. and 75 F. atmospheric temperatures are distinctly higher than are found in average commercial practice. Thus, the majority of automobile gasolines sold in the winter months not only have not been approaching in Reid vapor pressure the proposed schedule, but also relatively few of them have had 12-lb. Reid vapor pressures at all. This is illustrated by the following facts:

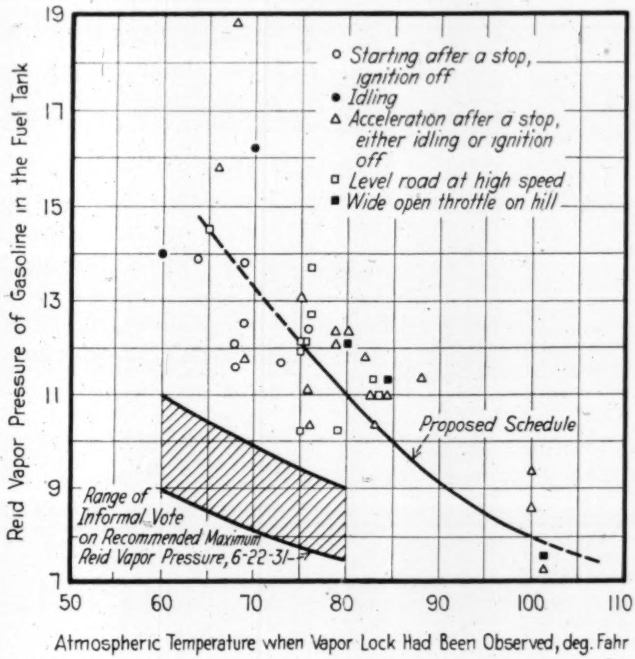

FIG. 2.—Reid Vapor Pressure *versus* Atmospheric Temperature for Vapor Lock 1931 to 1934 Cars.

1. In the surveys of winter gasolines made by the *Oil and Gas Journal* in 1930 to 1933(3) only 1 of the 131 gasolines included had a Reid vapor pressure of 12 lb., and the average Reid vapor pressure of the regular-price gasolines collected in the 1933 survey was 9.5 lb.

2. Among the gasolines of major distributors, as collected in Detroit in the month of February during the years 1932 to 1934 by General Motors Research Division, only 2 out of 66 had Reid vapor pressures as high as 12 lb. More than one-half of the regular-price and premium-price gasolines included in these surveys had Reid vapor pressures of 10 lb. or less.

3. Data given in the report presented to Technical Committee A recently by R. C. Alden(4) show that only 1 in 8 of the winter gasolines (collected in December, January, and February) had a Reid vapor pressure as high as 12 lb., and the average Reid vapor pressure of all the gasolines included in Alden's December to February survey in 1933 to 1934 was 10.5 lb.

Relationship of the Proposed Schedule to Observed Vapor Lock:

The fourth reason, and the most important of all, is that many cars now in use will give vapor lock on the gasolines named at the atmospheric temperatures which correspond to them in the proposed schedule. This is shown by Fig. 2, which is a chart of Reid vapor pressure plotted against the atmospheric temperatures at which vapor lock has been observed in our own experience. The data represent results obtained on cars of 1931 to 1934 models, chiefly the later models, and they include cars of all price classes. The nature of the vapor lock observed in each instance is designated. The heavy solid-line curve on the chart has been drawn through the points which represent the schedule of suitable Reid vapor pressures proposed by the Section on Vapor Lock, as given at the outset. That many of the points representing vapor lock lie within the region below this curve is apparent from the chart.

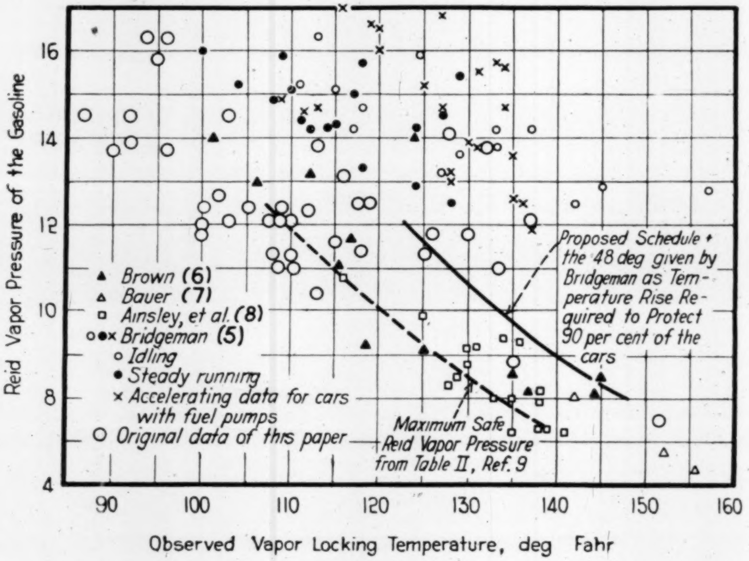

FIG. 3.—Reid Vapor Pressure *versus* Observed Vapor-Locking Temperature.

That the original data given on Fig. 2 are in general agreement with the results of other investigators, as presented in the literature, is shown by the points plotted on Fig. 3. Many of the published results of experimenters on vapor lock are not given in terms of atmospheric temperature but in terms of observed fuel temperature at the time, or at the point, of vapor lock. Consequently, they cannot be properly shown on a chart of atmospheric temperature, such as Fig. 2. The points plotted on Fig. 2 in terms of Reid vapor pressure *versus* atmospheric temperature for vapor lock are shown also on Fig. 3 in terms of Reid vapor pressure *versus* observed fuel temperature at the time of vapor lock; and, in addition, are plotted results of Brown(6), of Bauer(7), of Ainsley, Baker, and Phillips(8), and of Bridgeman(5). Recognizing the unavoidable variables involved, there seems to be a fair degree of agreement among the actual vapor locking temperatures observed by the different investigators. In the main, the data of Bridgeman lie above and to the right of that of the other investigators. It is believed that the reason for this is that Bridgeman's results were for the most part obtained at speeds not exceeding 40 m.p.h.

With further reference to Fig. 3, attention is called to the solid-line curve shown. This curve has been constructed by adding 48 deg. to each of the atmospheric temperatures named in the proposed vapor pressure schedule given above. The figure of 48 deg. is based upon the conclusion of Bridgeman(5) from his survey of the temperatures reached in the fuel systems of 1930, 1931, and 1932 cars that "in order to afford protection to more than 90 per cent of the cars on the road, a temperature rise of about 48 F. at constant speed must be assumed."

The lower or broken-line curve on Fig. 3 has been drawn from the data given in an early report of the Section on Vapor Lock as representing maximum safe Reid vapor pressure at 100 F.(9). And, as is apparent from the figure, this curve does come well toward the lower boundary of the points which represent observed vapor lock, when the results of the several investigators are taken into account. Mention may be made further of the fact that, compared on the horizontal scale, the broken-line curve lies 12 to 13 deg. below the solid curve at each given Reid vapor pressure. Compared on the vertical scale, the differences between the two curves amount to about 2 lb. Reid vapor pressure.

Also the broken-line curve on Fig. 3, which is in the lower portion of the band of observed vapor-locking conditions but still within it, lies about 35 deg. above the atmospheric temperatures named in the schedule of permissible Reid vapor pressures proposed by the Section on Vapor Lock. As of interest and apparent significance in this connection, note the following further quotation from Bridgeman(5) with regard to the *average* temperature rise in the fuel systems of cars during constant speed driving:

> "It appears therefore that during summer operation at constant speed driving under vapor-locking conditions, the temperature rise in that part of the fuel system most sensitive to vapor lock averages 34 F. above the atmospheric temperature."

Thus, on the basis of the comparison made in Fig. 3 and discussed above, it appears that the original vapor lock data of Fig. 2, which are presented directly in terms of the atmospheric temperatures at which vapor lock was experienced with gasolines of different Reid vapor pressures, are not out of line with those of other investigators. And the data of Fig. 2 show that many of the cars now in use will give vapor lock on the gasolines conforming to the schedule proposed by the Section on Vapor Lock.

An Alternative Suggestion:

In the light of the foregoing discussion, it is believed that the basis of the atmospheric temperatures given in the schedule of suitable Reid vapor pressures should be more precisely defined as to meaning, and that the temperatures used in the schedule should bear some relation to the temperature of the air in which cars are actually run. Such a schedule would give the gasoline marketer a sound basis of vapor pressures to work to, but it would allow him a suitable degree of freedom of choice in respect to actual marketing policies to take care of the usual variations in weather conditions which occur in each specific locality.

Putting the matter on this basis, it is believed that the following schedule of vapor pressures would be a suitable one:

TEMPERATURE OF AIR IN WHICH CAR IS DRIVEN, DEG. FAHR.	HIGHEST REID VAPOR PRESSURE OF GASOLINE IN FUEL TANK OF CAR GIVING FREEDOM FROM VAPOR LOCK IN MAJORITY OF CARS ON THE ROAD
95	8
80	10
65	12

Note that the atmospheric temperatures named above are those of the air in which the car is driven, and that the Reid vapor pressures are those of the gasoline in the fuel tank of the car, or as delivered from the filling station. The figures given are thus based directly upon the actual operation of cars, and they also relate directly to the experimental data as obtained. The above schedule provides for a greater degree of freedom from vapor lock at the lower temperatures than at the high. This is thought to be desirable both because more driving is done at such temperatures and because vapor lock when it does occur at the lower temperatures is much more disturbing to drivers of cars than when it occurs on very hot days. Naturally the schedule as given would need to be modified accordingly if any other form of temperature or vapor pressure concept than that upon which it is based were introduced.

Improvements Made in Cars:

In stating the objections to the schedule of Reid vapor pressures proposed by the Section on Vapor Lock, as has been done above, it should be said that the obligation and the desire of the automobile industry to make cars as free as possible from vapor lock is not lost sight of. Cars have been improved in this respect during recent years, and still further improvement can and will be made. In considering the proposed schedule, however, the guide must be in large measure the characteristics of existing cars.

TABLE I.—CHANGES IN THE MAXIMUM TEMPERATURE RISES IN THE FUEL SYSTEMS OF FIVE REPRESENTATIVE CARS OVER A 5-YEAR PERIOD. Showing an average reduction of about 15 deg. Fahr., which corresponds in turn to a boost of approximately 2 lb. in permissible Reid vapor pressure.

Car	Maximum Temperature Rise of Fuel Systems Above Atmospheric, for Car Speed of 60 m.p.h., deg. Fahr.					
	1929 Cars		1931 Cars		1934 Cars	
	Steady Driving	Idling Afterwards	Steady Driving	Idling Afterwards	Steady Driving	Idling Afterwards
No. 1	29	67	37	65	25	38
No. 2	45	61	50	69	42	55
No. 3	57	81	52	67	27	48
No. 4	58	85	..	..	38	85
No. 5	..	..	44	66	31	60
Average	47	73	46	66	33	57

The improvement which has already been made in cars from the viewpoint of freedom from vapor lock, as just mentioned, has extended in two directions: first, in the reduction of fuel temperatures at the carburetor; and, second, in the enlargement of vapor-handling capacities. The first of these which, because it prevents loss of vapor, is recognized as the more desirable of the two, is illustrated by the data of Table I. Presented there are figures on the temperature rises in the fuel systems of the five representative cars, including all price classes, for which comparable temperature data happen to be available.

Note that over the 5-year period, 1929 to 1934, the average reductions in the maximum temperatures reached by the liquid gasoline in the fuel systems of these five cars were as follows: for steady driving at 60 m.p.h., 14 deg. Fahr.; for idling after driving at 60 m.p.h., 16 deg. Fahr. These reductions in temperature are considerable, for they have been made in spite of increased engine sizes and correspondingly larger amounts of heat to be dissipated.

As an instance of the improvement which consists in enlarging vapor-handling capacities through modified design may be cited the case of one car on which there are comparative data. This car was one that when the vapor pressure of the gasoline was too high suffered from the form of vapor lock which reduced its driving speed. By a modification of design the vapor-handling capacity of this car under high-speed driving conditions was raised from less than 5 volumes of vapor per one volume of liquid gasoline at the point of vapor lock to 20 volumes, which latter is a very high vapor tolerance indeed to exist at the time when fuel demand is close to a maximum.

With further reference to the improvement made in cars in recent years with regard to freedom from vapor lock, attention is called to the shaded area at the lower left of Fig. 2. The top and bottom boundaries of that area represent the upper and lower limits of the "Recommended Maximum Reid Vapor Pressure" at the corresponding atmospheric temperatures, as based upon the 17 informal votes of men who attended a meeting of the Section on Vapor Lock on June 22, 1931, at which the vote was taken (9). Note that the upper boundary of the consensus of opinion at that time on the proper Reid vapor pressures to be specified lies below the points on the chart representing observed vapor lock on recent cars. Note further that the most optimistic votes then were, at each atmospheric temperature, for Reid vapor pressures of 2 lb. or more below the schedule now proposed by the Section on Vapor Lock.

It is the impression of some that it would be a simple matter for the car designer to so arrange, that vapor lock could not occur on any gasoline. This impression is based upon the comparatively small temperature rise which occurs during steady driving conditions in cars having the smaller power plants. But, in this connection, mention may be made of the fact that vapor lock quite commonly occurs just following the time when the car has been standing still for a period, either with the engine idling or stopped. That this is so may be seen from the data of Fig. 2, which shows that vapor lock frequently occurs when attempting to start the engine after a stop with ignition off, or else during acceleration after such a stop or after idling for a period. No matter how well protected and cooled a fuel system may be from the viewpoint of conditions prevailing while the car is moving through the air, the gasoline in the carburetor, in the fuel pump, and in the fuel lines under the hood will tend to approach the temperature which prevails under the hood when the car is standing. And that temperature is determined not only by the high temperature of the exhaust manifold, which is sometimes very hot indeed, but also by the temperature of the cooling medium which latter is usually from 140 to 180 F. The approach of the gasoline to the equilibrium temperature reached under the hood is a thing which, as experience has shown, can be retarded somewhat, but which it is very difficult to prevent. It is in spite of these fundamental obstacles that the improvement cited above has been made and that any further improvement must be effected.

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(1) C. K. Francis, "Method for Determining Vapor Lock," *Oil and Gas Journal* March 17, 1932, p. 22.

(2) O. C. Bridgeman, "The Effect of Automobile Design on Vapor Lock," *Proceedings*, Am. Petroleum Inst., Section III, Vol. 14, November, 1933, p. 18.

(3) W. T. Ziegenhain, "Average Octane Number of the Regular Grade Gasolines Increased Sharply in Past Year," *Oil and Gas Journal*, December 21, 1933, p. 8.

(4) R. C. Alden, "The Status of the Vapor Lock Problem," a report presented to Technical Committee A on Gasoline of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants, March, 1934.

(5) O. C. Bridgeman, H. S. White, and F. B. Gary, "The Present Status of the Vapor Lock Problem," Appendix F, Cooperative Fuel Research Committee Minutes, September 18, 1933.

(6) G. G. Brown, "The Volatility of Motor Fuels," *Engineering Research Bulletin, No. 14*, University of Michigan, May, 1930, p. 274.

(7) W. C. Bauer, "Vapor-Locking Tendencies of Automotive Fuel Systems," *Journal*, Soc. Automotive Engrs., June, 1930, p. 736.

(8) W. G. Ainsley, L. E. Baker, and E. B. Phillips, "Methods of Testing Automobiles for Potential Vapor Lock," *Proceedings*, Am. Petroleum Inst., Section III, Vol. 14, November, 1933, p. 22.

(9) Report of Section III on Vapor Lock to Technical Committee A on Gasoline of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants, October 31, 1931.

[For Discussion on Vapor Lock, see page 560.—ED.]

MOTOR FUELS AND VAPOR LOCK

BY GEORGE GRANGER BROWN[1]

SYNOPSIS

All of the available actual road-test data taken under vapor-locking conditions, on the vapor pressure of fuel in the tank of car required to cause vapor lock, have been plotted and compared with the limits recommended by the Section on Vapor Lock. Under steady driving conditions tests on 60 cars indicate that only 3 might be expected to experience difficulty, and that 56 would be free from vapor lock even under the maximum temperature conditions allowed in the recommended limits. Upon idling after hard driving, tests on 56 cars indicate that 7 might vapor lock and that 48 would be entirely free from this difficulty. The recommended limits are not intended to prevent vapor lock entirely, but to be a reasonable compromise for present conditions. They have been found to agree with present commercial practice and are somewhat more conservative at high temperatures. If all cars were as good as the best, the vapor pressure limits could be raised about 4 lb. with less vapor lock difficulty than is now experienced on some cars. The recommended limits therefore insure reasonable protection to the user from vapor lock so far as the fuel is concerned.

Vaporization of the gasoline in fuel lines, pumps or carburetor passages designed to carry and meter liquid fuel restricts the normal flow of fuel. Vapor lock is said to occur when this restriction in flow of fuel is sufficiently pronounced to interfere with the normal operation of the motor. In severe cases the motor may cease functioning entirely.

The cause of vapor lock in motor cars is primarily the overheating of fuel lines, pumps and carburetors. If the fuel were not heated in its flow to the carburetor above its temperature in the supply tank on the vehicle there would be no vapor lock. Unfortunately this most desirable condition does not exist and the vaporizing tendency or vapor pressure of the fuel as it becomes heated to high temperatures in the fuel lines of the vehicle must be limited according to the vapor-handling capacities and temperatures of these fuel systems if vapor lock is to be prevented.

Vapor lock has been recognized for many years and carefully studied during the past eight years by numerous investigators with the accumulation of rather complete data on the vapor-locking characteristics of the cars now on the road. On July 18, 1930, the

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Section on Vapor Lock, of Technical Committee A of the Society's Committee D-2 on Petroleum Products and Lubricants was appointed *"to ascertain by engine test data what quality in gasoline is necessary to give reasonable protection to the user so far as the fuel is concerned;*

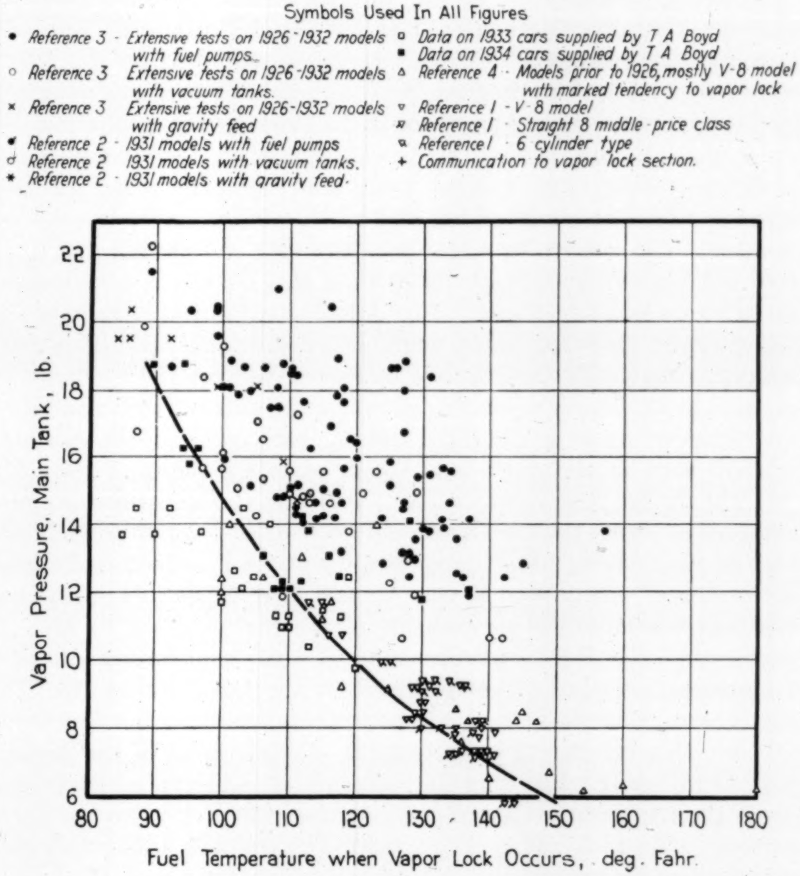

FIG. 1.—Data on the Reid Vapor Pressure at 100 F. of the Fuel in the Main Tank of the Car, and the Fuel-Line Temperature at Point of Vapor Lock. Both sample of fuel and fuel-line temperature taken at time of vapor lock.

then to specify the method by which this quality should be determined in inspection test, and finally, to set the proper limits to be included in specifications for gasoline."

The data plotted in the accompanying figures were collected for this committee from published work, and unpublished data reported to and obtained by members of the Section on Vapor Lock. It is believed that all available reliable engine data on vapor lock are included.

Vapor Pressure Test Method:

Careful consideration of the various tests available indicated that the Tentative Method of Test for Vapor Pressure of Natural Gasoline (Reid Method) (D 323 - 32 T)[1] appeared to be the most satisfactory method for determining the vaporizing tendency of gasoline to give reasonable protection to the user so far as the fuel is concerned. Because of the different characteristics of the fuel systems of different cars, some systems being able to handle much greater volumes of vapor than others, and to the differences in operating conditions encountered in actual use, it cannot be expected that any test will accurately indicate the temperature at which a fuel may cause vapor lock on all cars. The data plotted in Fig. 1 include all the available data(**1**,**3**,**4**)[2] taken under actual operating conditions and indicate a reasonably satisfactory agreement between the Reid vapor pressure at 100 F. of the gasoline in the supply tank of the car and the fuel temperature at which vapor lock is observed. A table showing symbols used in all figures is included, preceding Fig. 1.

The dashed line shown in Fig. 1 indicates the approximate temperature at which the gasoline would exert a Reid vapor pressure of 14.7 lb. when having a Reid vapor pressure at 100 F. as indicated along the ordinate. In general it may be said that vapor lock might be expected whenever the temperature of the fuel is raised to such a degree that the Reid vapor pressure at that temperature exceeds atmospheric pressure. Practically all of the test data reported in Fig. 1 were obtained with atmospheric pressures somewhat less than 14.7 lb., although run at low altitudes. Even so most of the points, particularly those taken from the most careful and complete survey(**3**), lie to the right of the dashed line because of loss in vapor pressure between main tank and point of vapor lock and the ability of most cars to handle appreciable quantities of vapor without difficulty, indicating that the above statement is conservative in being on the safe side.

Any errors or carelessness in sampling tend to show a low vapor pressure and would make the point lie lower than it should in Figs. 1 and 3. Errors in reading the proper temperature in the fuel line such as reading the temperature of pump inlet when vapor lock

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 765 (1932); also 1933 Book of A.S.T.M. Tentative Standards, p. 569.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 559.

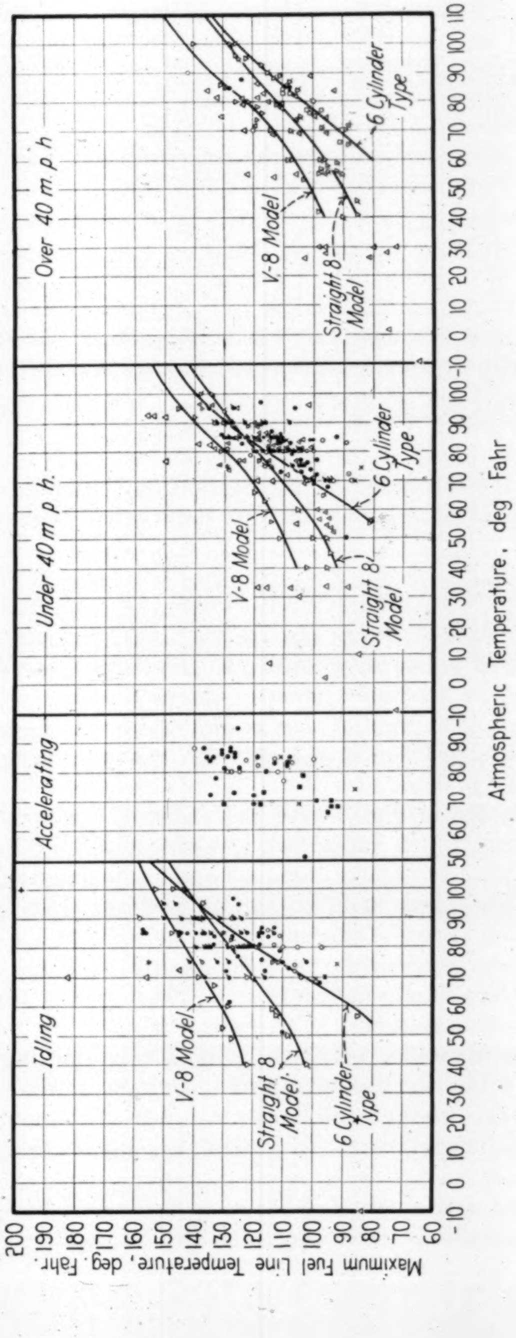

FIG. 2.—Maximum or Vapor-Locking Fuel-Line Temperatures Observed at Different Atmospheric Temperatures When Idling Motor After Driving, Accelerating After Idling, Driving at Constant Speeds not Exceeding 40 m.p.h., and Driving at Constant Speeds Exceeding 40 m.p.h.

occurs at a higher temperature in the pump, misplace the point horizontally in Fig. 1 and vertically in Fig. 2 but introduce no direct error in other figures.

Point of Sampling:

Changes in the vapor pressure characteristics of a gasoline take place in the supply tank of the car after filling, and in the fuel system after leaving the supply tank(1,3,4) as well as in shipments of motor fuel from the refinery to the service station. For these reasons the most logical place to sample the gasoline for determining its vapor pressure to insure reasonable freedom from vapor lock is at a point nearest the ultimate consumption. It is impractical to attempt to sample the gasoline for specification purposes after it is delivered into the supply tank of the ultimate consumer. The closest practical approach to the point of ultimate consumption is the gasoline as delivered from the pump at the service station, and this is the point which has been recommended by the Section on Vapor Lock as the proper point for sampling motor fuel for Reid vapor pressure determination.

Basis for Recommended Vapor-Pressure Limits:

Although the data plotted in Fig. 1 might lead to recommendations limiting Reid vapor pressures according to the temperature to which the fuel is heated in the fuel system of the motor car, such recommendations are of little practical use as only those who have investigated the vapor-lock problem have definite ideas as to the temperatures reached in the fuel system.

The data plotted in Fig. 2 give the observed fuel temperature under conditions at which vapor lock was observed for different atmospheric temperatures and for different operating conditions. The inverted pyramids(1) and modified circles and crosses(2) are data obtained under non-vapor-locking conditions but indicate the observed maximum fuel line temperature as a function of atmospheric temperature for the conditions indicated. All other points were taken under vapor-locking conditions.

It is obvious from Fig. 2 that the critical vapor-locking temperatures reached in the fuel line, pump or carburetor on different cars and under different driving conditions vary over wide limits for the same atmospheric temperature. In general, the highest temperatures in the fuel lines are reached under idling conditions, particularly when the engine is idled after a period of heavy or high-speed driving. For steady driving conditions lower maximum fuel line temperatures are reached when driving at high speeds, over 40 miles per hour, than when driving at lower speeds. The greater tendency of some cars to vapor-lock under conditions of high-speed driving is therefore due, not to higher fuel line temperatures, but to the greater fuel requirements of the engine which decrease the reserve capacity of the fuel pump at high speeds.

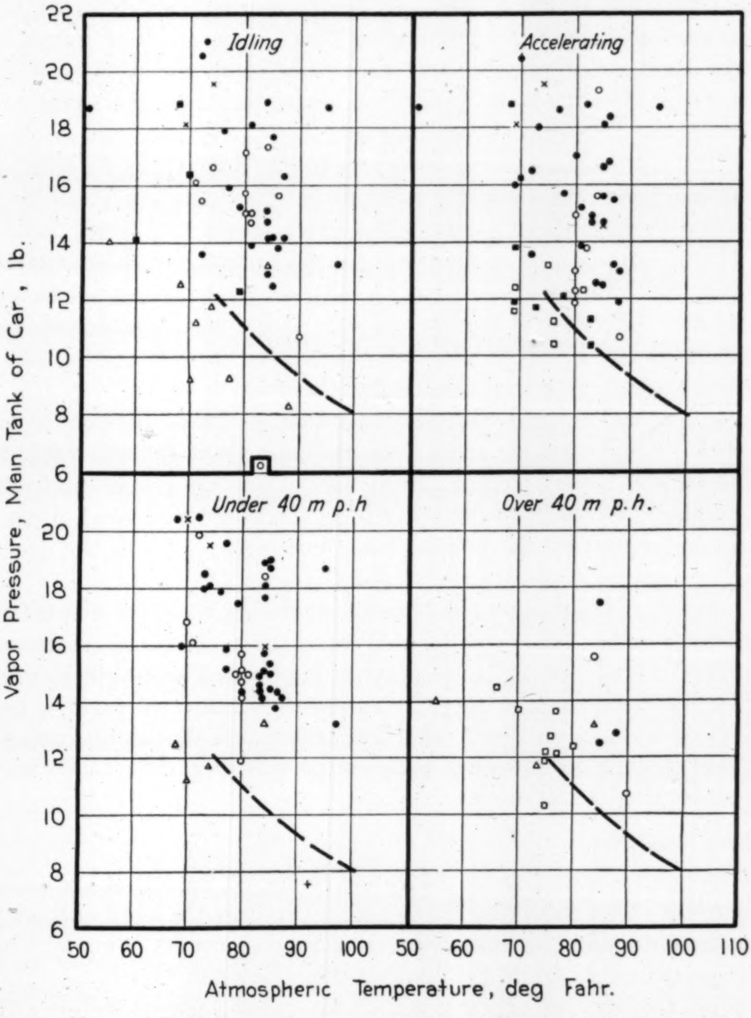

FIG. 3.—Observed Reid Vapor Pressure at 100 F. of Fuel in Main Tank of Car at Time of Vapor Lock as Related to Atmospheric Temperature When Idling Motor After Driving, Accelerating After Idling, Driving at Constant Speeds not Exceeding 40 m.p.h., and Driving at Constant Speeds Exceeding 40 m.p.h.

The discrepancies in fuel-line temperatures between different cars, and under different driving conditions, for the same atmospheric temperature are greater than the discrepancies between the Reid vapor pressure at 100 F. and fuel temperature at which vapor lock occurs, as given in Fig. 1, indicating that the accuracy or suitability of the Reid vapor pressure test as a basis of specification to insure reasonable freedom from vapor lock is better than the agreement between the different cars on the road.

The data plotted in Fig. 3 include all the available data obtained in actual engine tests under conditions of actual operation and under vapor-locking conditions, giving the vapor pressure at 100 F. of the fuel in the supply tank of the car at the time of vapor lock as observed under the indicated operating conditions and atmospheric temperature. The dashed line corresponds to the maximum Reid vapor pressure at 100 F. of the gasoline as delivered from the pump at the service station as recommended by the Section on Vapor Lock to insure reasonable freedom from vapor lock so far as the fuel is concerned. In interpreting these data, it is clear that the recommended limits would insure freedom from vapor lock under the maximum atmospheric temperature and operating conditions indicated, in those cars represented by the points above and to the right of the dashed line.

Points computed on the basis of maximum fuel-line temperature observed under non-vapor-locking conditions combined with data obtained on independent tests as to the vapor pressure at 100 F. which will cause vapor lock at these maximum fuel-line temperatures, have not been included in Fig. 3, as it has been found that data computed in this manner do not agree with actual road test data taken under vapor-locking conditions(3,4).

Idling Conditions:

All of the data points located below and to the left of the dashed line in the idling section of Fig. 3 were taken from the work of Brown(4) and were obtained on an 8-cylinder, V-type motor, 1922 model which was particularly bad in its vapor-locking characteristics.

Of the 42 cars actually tested for vapor lock under operating conditions when idling after a period of hard driving, 41 would be free from vapor lock, and one might be expected to experience difficulty when using fuels of the maximum Reid vapor pressure and at the maximum atmospheric temperature within the limits recommended by the Section on Vapor Lock.

Computed points from the data plotted in Figs. 1 and 2 as obtained by Ainsley, Baker and Phillips(1), on the 8-cylinder, V-type motor, would indicate that this car which is also particularly sensitive to vapor lock under idling conditions would also experience difficulty. But the data are not included in Fig. 3 because they are not direct experimental data but are computed from two separate tests.

Accelerating After Idling:

Of the 52 cars tested when accelerating after idling (Fig. 3), 5 points supplied by T. A. Boyd fall below the line, and 45 points lie above the line, indicating that under these conditions of operation, vapor lock might be expected on 5 cars out of the 52 tested when using fuels of the maximum Reid vapor pressure at the maximum atmospheric temperature within the limits recommended by the Section on Vapor Lock.

Constant Speed Below 40 Miles per Hour:

Of the 51 cars tested at constant speeds up to 40 miles per hour (Fig. 3), 49 cars would be entirely free from vapor lock and 2 cars, the same 8-cylinder, V-type motor mentioned above and another outmoded car privately reported, might experience difficulty with fuels of the maximum vapor pressure at the maximum atmospheric temperature within the limits recommended by the Section on Vapor Lock.

Constant Speed Over 40 Miles per Hour:

When driving at high speeds (over 40 m.p.h.) the data taken on 17 cars (Fig. 3) indicate that those cars with the worst vapor-locking characteristics under idling conditions would be reasonably free from vapor lock, and that 14 of the 17 cars would be entirely free from vapor lock with fuels of the maximum vapor pressure within the limits recommended by the Section on Vapor Lock. Only one point reported by T. A. Boyd is decidedly below the line.

Recommendations of Section on Vapor Lock:

It is a practical impossibility to insure absolute freedom from vapor lock in all cars under all conditions of service and yet furnish fuels which will give satisfactory starting and acceleration. Clearly a compromise must be effected. This is indicated by the charge to the Section on Vapor Lock to recommend limits and a method of test that would insure "reasonable protection to the user so far as the fuel is concerned." This might be taken to indicate that the committee should confine its attention to those cars which are reasonably good in their vapor-locking characteristics and set limits which will insure freedom from vapor lock on these cars only. Instead the Section has considered practically all cars on the road, those excessively bad in vapor-locking characteristics as well as those of reasonable design. The complete data have been included in this paper and presented in the various figures.

In recommending new specifications which have not been previously in effect, it is always desirable to adopt limits which closely approximate present commercial practice. This point has received careful consideration in the following limits recommended by the Section on Vapor Lock to insure reasonable protection to the user so far as the fuel is concerned:

> "That the Reid vapor pressure (A.S.T.M. Method D 323 – 32 T) of fuels as delivered from the service station pump or to the fuel tank of the vehicle for use at maximum atmospheric temperature of 100 F. should not exceed 8 lb., at a maximum atmospheric temperature of 85 F. should not exceed 10 lb., and at a maximum atmospheric temperature of 75 F. should not exceed 12 lb."

It is clear that if all the cars on the road were of as good design in regard to vapor-locking characteristics as the average of the best cars that have been tested, these vapor-pressure limits could be raised about 3 to 4 lb., corresponding to 11 lb. for a maximum atmospheric temperature of 100 F., 14 lb. for a maximum atmospheric temperature of 85 F., 16 lb. for a maximum temperature of 75 F. and about 19 lb. for a maximum temperature of 65 F. and insure entire freedom from vapor lock under all normal conditions. Perhaps such good design in all cars is not to be expected immediately, but we should be able to expect that all cars will improve their fuel systems so that the above limits will insure reasonable freedom from vapor lock in so far as the fuel is concerned, soon after the cars now in service are retired. At least these limits just mentioned should be our immediate object as they will in fact insure reasonable freedom from vapor lock *so far as the fuel is concerned*, that is, reasonable freedom from vapor lock in all cars as good as the average of the best cars now on the road.

For the immediate present, the recommended limits of 8 lb. for a maximum atmospheric temperature of 100 F., 10 lb. for a maximum atmospheric temperature of 85 F., and 12 lb. for a maximum atmospheric temperature of 75 F. will insure reasonable freedom from vapor lock under all ordinary driving conditions including high-speed operation. Some difficulty is to be expected on a limited number of cars which are particularly bad offenders in regard to vapor lock whenever the speed is reduced, or the motor is idled, after sustained high-speed driving or heavy pulling. But it must be emphasized that no more difficulty will be experienced when using fuels of these recommended vapor-pressure limits than is now actually experienced in operation, because the recommended vapor-pressure limits are in close agreement with present commercial usage.

A careful and complete survey covering the entire country(5) showed these recommendations to be closely representative of commercial practice except that present commercial practice allowed an average vapor pressure of 8 lb. under conditions where the maximum temperature is over 105 F. This is probably why vapor lock is confined largely to warm weather and popularly classed as a hot-weather complaint.

Conclusion:

Adoption of the recommended vapor-pressure limits will insure reasonable protection from vapor lock so far as the fuel is concerned. The recommended limits insure the same freedom from vapor lock as is now furnished the car user except in extremely warm weather when greater protection will be insured by the adoption of these limits.

Improvement in motor-car design to the extent that all cars are as good as the best would allow an increase of about 4 lb. in these limits.

REFERENCES

(1) W. G. Ainsley, L. E. Baker and E. B. Phillips, "Methods of Testing Automobiles for Potential Vapor Lock," *Proceedings*, Am. Petroleum Inst., Section III, Vol. 14, November, 1933, p. 22. (3 cars.)

(2) O. C. Bridgeman, H. S. White and F. B. Gary, "Gasoline Vapor Pressures Permissible with 1931 Motor Cars," *Proceedings*, Am. Petroleum Inst., Section III, Twelfth Annual Meeting, December, 1931, p. 40. (47 cars.)

(3) O. C. Bridgeman, H. S. White and F. B. Gary, "The Present Status of the Vapor-Lock Problem," *Proceedings*, Natural Gasoline Assn. of America, Twelfth Annual Convention, Tulsa, May, 1933, p. 74. (51 cars.)

(4) G. G. Brown, "The Volatility of Motor Fuels," *Engineering Research Bulletin No. 14*, University of Michigan, May, 1930, p. 274. (4 cars.)

(5) W. Mendius and W. G. Ainsley, "Conservation of Volatile Hydrocarbons," Fourth Mid-Year Meeting, American Petroleum Institute, Pittsburgh, May 24, 1934.

DISCUSSION ON VAPOR LOCK

MR. R. C. ALDEN[1] (*presented in written form*).—Mendius and Ainsley[2] have demonstrated that if all cars were equivalent to the best car (from the vapor lock viewpoint) there could be incorporated in motor fuels, a billion gallons annually of light hydrocarbons now wasted, with absolute freedom from vapor lock under the highest temperature conditions ever recorded.

Alden[3] has shown that butane is the marginal hydrocarbon in the vapor lock problem. There is enough butane available to constitute 20 per cent of the motor fuel supply. Each per cent of butane added to motor fuel would result in annual refinery savings of about $55,000,000 by virtue of lessened requirements for crude oil and reduced ethylizing costs.

The subject of vapor lock is not a simple one. Every factor involved is open to several definitions and is seldom rigorously defined. As examples, consider the following terms:

1. *Reid Vapor Pressure.*—This may be measured at the refinery, filling station, in the automobile fuel tank or at the actual point of vapor lock. Obviously the vapor pressure which causes vapor lock is the vapor pressure of the gasoline at the point of vapor lock. However, vapor pressure at this point is extremely difficult of measurement and use has been made by Bridgeman[4] of the more practical determination of the vapor pressure in the automobile fuel tank.

2. *Fuel Line Temperatures.*—These may be determined in laboratory apparatus or in the fuel lines of automobiles under non-vapor-locking or under vapor-locking conditions. The temperatures obtained in simple laboratory equipment are seldom comparable to those occurring in the variable fuel systems in use. Temperatures obtained in automobile fuel lines under non-vapor-locking conditions are generally higher than those at which vapor lock occurs. This is because temperatures are reduced as the vapor-locking condition is approached due to the cooling effect of increased vaporization. Vapor-locking fuel line temperatures should be the fuel line tempera-

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[1] Director of Research, Phillips Petroleum Co., Bartlesville, Okla.

[2] W. Mendius and W. G. Ainsley, "Conservation of Volatile Gasoline Materials," *Proceedings*, Fourth Mid-Year Meeting, Am. Petroleum Inst., Vol. 15, Section III, p. 68.

[3] R. C. Alden, "Butane—A Wasted Asset of the Petroleum Industry," *Proceedings*, Fourth Mid-Year Meeting, Am. Petroleum Inst., Vol. 15, Section III, p. 74.

[4] O. C. Bridgeman, H. S. White and F. B. Gary, "Vapor Handling Capacity of Automobile Fuel Systems," *Journal*, Soc. Automotive Engrs., Vol. 32, No. 4, April, 1933, p. 157.

tures at the time and point of actual vapor lock in actual automobiles and under specific driving conditions. In view of the lack of established relationships between non-vapor-locking and vapor-locking fuel line temperatures these determinations have little significance at present. The important variable is the vapor-locking Reid vapor pressure.

3. *Point of Vapor Lock.*—The point of highest temperature or lowest pressure in the fuel system is not necessarily the point of vapor lock. Such factors as removal of vapors by the carburetor bowl, increase in pressure by a fuel pump, or an increase in the vapor handling capacity of subsequent portions of the fuel system invalidate such a conclusion.

4. *Atmospheric Temperature.*—It is a simple matter to determine the atmospheric temperature at the time of vapor lock, but a definite problem is introduced when attempts are made to include this factor in generic specifications for gasoline. The following temperature terms have been variously used in vapor lock discussions: absolute monthly (or seasonable) maximum, average monthly (or seasonal) maximum, average daily maximum (by months or seasons) and mean temperatures. As a specification item the writer prefers the highest temperature basis. It seems better to say that a seasonal gasoline is specified, for example, of 8-lb. vapor pressure to insure reasonable freedom from vapor lock for a maximum atmospheric temperature of 105 F. than for an average daily maximum temperature of 85 F. Although the two definitions would be equivalent, under the latter one, whenever the atmospheric temperature exceeded 85 F., too many people would be expecting vapor lock and looking for trouble.

The Section on Vapor Lock, of Technical Committee A of the Society's Committee D-2 on Petroleum Products and Lubricants, has made no attempt to correlate climatic conditions with the recommendations it has made. The specifications, as recently promulgated, simply state the vapor pressure limits of gasoline which will give reasonable freedom from vapor lock when used at or below the atmospheric temperatures named. Presumably the interpretation of these specifications would depend on the purchasers and sellers of gasolines. If more definite specifications are considered necessary, this is a task for the future.

As will be shown later (Fig. 2) it may be important to define "atmospheric temperature" in relation to climate because if the unmodified term is rigorously applied to climatic atmospheric temperatures the committee's recommendations fall far short of describing present-day commercial practice. The desirability of taking this step will depend on the wishes of consumers and manufacturers of gasoline.

5. *Vapor Lock.*—In respect to vapor lock itself there are many kinds of vapor locking. The varieties most important to the motorist are the ones which cause him the most inconvenience, such as engine stoppage on idling after a hard run, or inability to start the engine after short stops following fast driving. The varieties of most concern to the automotive engineers are those which lead to loss of power and poor fuel economy.

This brief review clearly shows that the subject of vapor lock is far from being thoroughly understood. The only simple thing about the problem is the cure. *The cure is to rationalize the fuel systems of a few car models to provide for the quality of gasoline which will most certainly be the economic supply of the immediate future.* This simple and inexpensive cure will accomplish so much improvement that the fine points of the phenomenon of vapor lock need not be investigated.

As early as 1926 we formulated seasonal and geographic specifications for vapor pressure. These specifications were established almost entirely from a study of then existing commercial practices, chiefly among the small independent marketers because it was there we found the extremes in gasoline qualities. The specifications were well within the extremes in gasoline qualities.

These specifications were put into practice in 1927 and except for minor refinements are still unchanged. A considerable gallonage has been and is being marketed. The following were the vapor pressure specifications:

MAXIMUM REID VAPOR PRESSURE AT REFINERY	MEAN MEAN TEMPERATURE, DEG. FAHR., FOR MONTH FOR POINT OF SHIPMENT (Nov. 1, 1928)	MEAN MAX. TEMPERATURE, DEG. FAHR. FOR MONTH FOR POINT OF SHIPMENT (Nov. 15, 1930)
13.00	10 – 25	Below 45
11.75	26 – 41	45 – 65
10.50	42 – 57	65 – 75
9.25	58 – 73	75 – 85
8.00	74 – 89	Above 85

Neither the actual commercial results obtained under the above and similar shipping schedules nor the experience of gasoline marketers as judged from extensive surveys of the Reid vapor pressure of gasoline at filling stations coincided with the conclusions to be derived from all the work up to and including the vapor lock study[1] of 1931 cars. It was noteworthy that with limited exceptions[2] none of the vapor lock work had been done under conditions of actual vapor lock.

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[1] O. C. Bridgeman, H. S. White and F. B. Gary, "Gasoline Vapor Pressures Permissible with 1931 Motors," *Proceedings*, Twelfth Annual Meeting, Am. Petroleum Inst., Section III, November, 1931, p. 40.

[2] G. G. Brown, "The Volatility of Motor Fuels," *Engineering Bulletin No. 14*, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

At this juncture the Natural Gasoline Association of America requested that future work should be under actual vapor-locking conditions in actual cars. As a result, the 1932 work was done under the requested circumstances. Additionally, enough cars to represent 90 per cent of the cars on the road were tested, the point of vapor lock was ascertained, sampling was soundly done, and extreme qualities in gasoline were investigated.

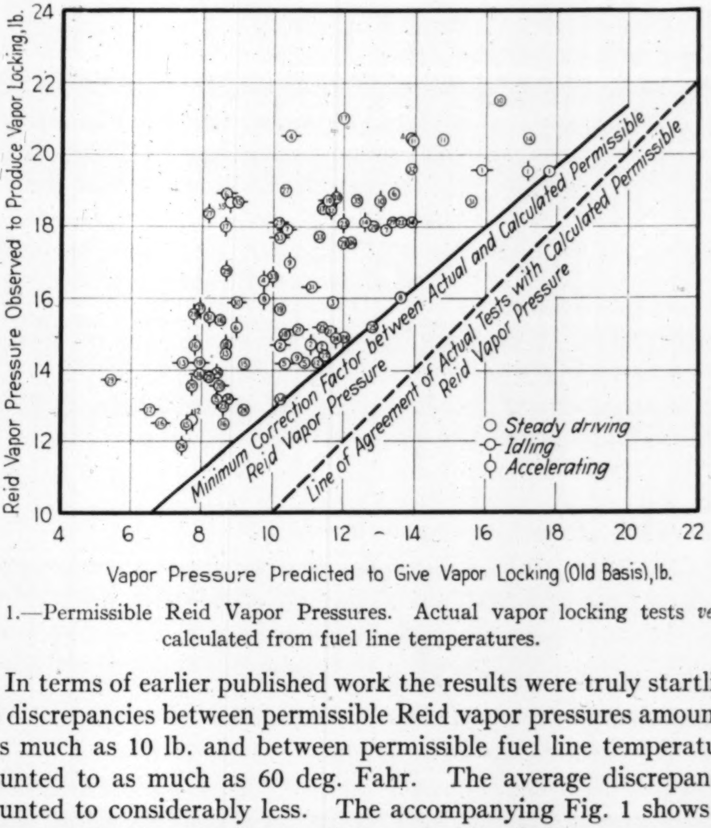

FIG. 1.—Permissible Reid Vapor Pressures. Actual vapor locking tests *versus* calculated from fuel line temperatures.

In terms of earlier published work the results were truly startling. The discrepancies between permissible Reid vapor pressures amounted to as much as 10 lb. and between permissible fuel line temperatures amounted to as much as 60 deg. Fahr. The average discrepancies amounted to considerably less. The accompanying Fig. 1 shows for the cars studied in 1932 the actual vapor locking Reid vapor pressures as compared with the permissible Reid vapor pressure calculated[1] according to the method used in the earlier C.F.R. work. The vacuum tank cars were omitted from Fig. 1 because they comprise a special case and would unnecessarily complicate the relationship presented.

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[1] O. C. Bridgeman, H. S. White and F. B. Gary, "Gasoline Vapor Pressures Permissible with 1931 Motors," *Proceedings*, Twelfth Annual Meeting, Am. Petroleum Inst., Section III, Novermber, 1931, p. 40.

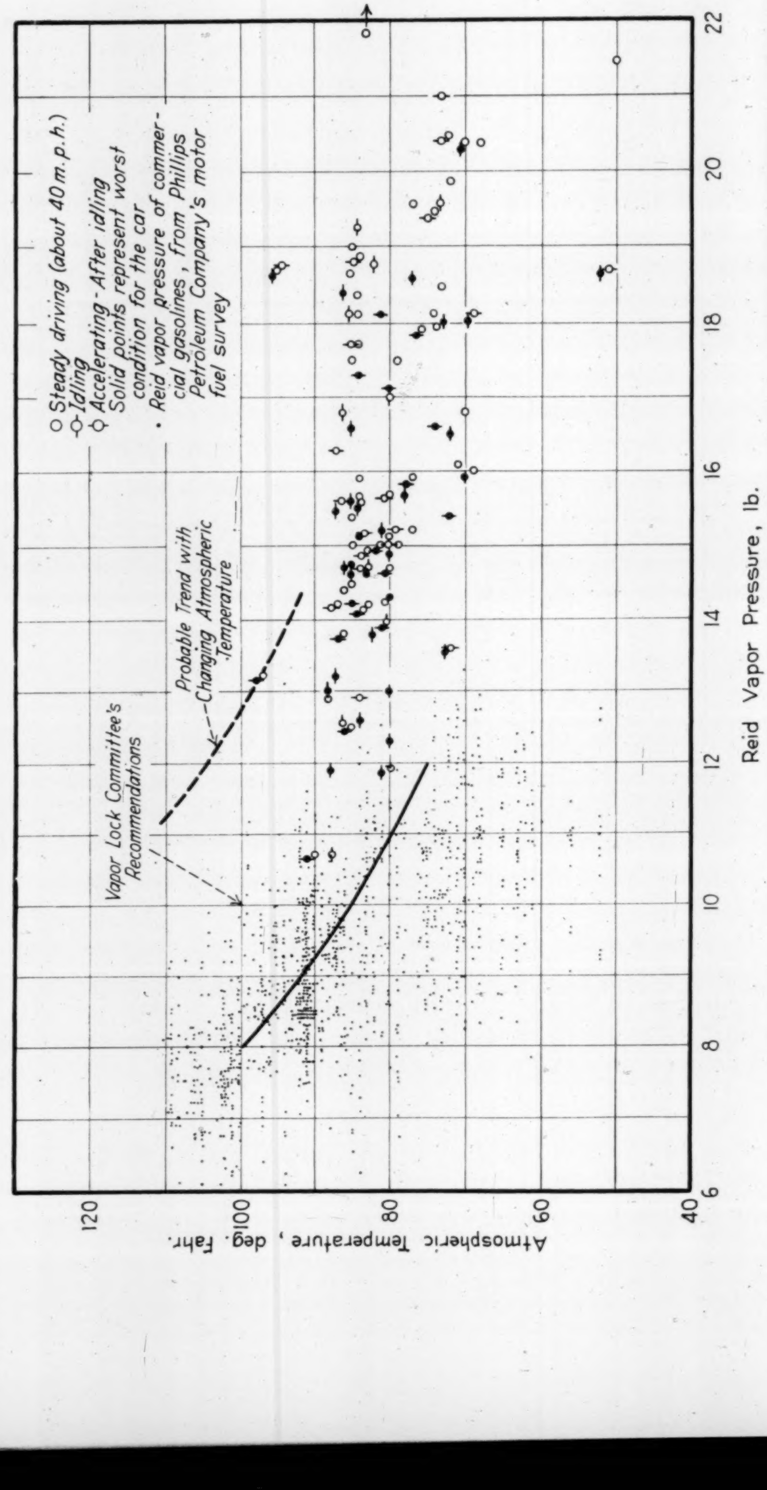

FIG. 2.—Permissible Reid Vapor Pressure in Cars *versus* Reid Vapor Pressure of Commercial Gasolines.

The fuel line temperatures used in Fig. 1 as a basis for the calculated permissible Reid vapor pressure are *vapor locking fuel line temperatures* which are probably several degrees lower than *non-vapor-locking fuel line temperatures*. Hence, many of the points in Fig. 1 should be moved to the left of the chart by almost a pound. By disregarding this factor there are still substantial increases in permissible Reid vapor pressure. The highest calculated permissible Reid vapor pressure in the study[1] of 1931 cars was 15 lb. From Fig. 1 it is to be noted that 57 of the 90 points are above 15 lb.

The full significance of Bridgeman's most recent work does not strike home until some relationship as in Fig. 1 is visualized. Old ideas based on *calculated* permissible Reid vapor pressure still persist. Thus Mendius and Ainsley in their recent A.P.I. paper used best car performance in terms of the old ideas and concluded that a mere billion of gallons of now wasted butane could be added annually to the motor fuel supply. The statement went unchallenged. As a matter of fact, *best car performance in the light of these new and rational investigations could create a market for not only all the butane currently extracted, but for all the butane available.*

There is another important relationship in the vapor lock problem which has not been shown before. The accompanying Fig. 2 shows the vapor locking Reid vapor pressures in 90 per cent of the cars on the road as determined by Bridgeman by actual experiment and the Reid vapor pressures of gasolines taken from filling station hoses. Both factors are plotted against atmospheric temperatures. In the case of the vapor locking Reid vapor pressures of automobiles, the atmospheric temperature is that of the individual test. In the case of the gasolines, the atmospheric temperature is the maximum ever recorded at the place where the sample was taken for the month of the year in which the sample was taken. The points representing cars may be considered as lines sloping sharply downward from left to right at angles depending on the heating and cooling characteristics of the individual cars represented by the points. Bridgeman tested[2] seven cars for heat input to the fuel system over a range of 45 deg. Fahr. in atmospheric temperature and found that with one exception the heat input declined very slightly with increasing atmospheric temperatures.

It is difficult to reconcile opinions which are based on earlier methods of attack with the conclusions as outlined in the preceding paragraph, which are derived from Bridgeman's recent work. The purpose of presenting Fig. 2 is to demonstrate that these new conclusions, based on actual vapor lock tests, agree much better with the experience of gasoline marketers.

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[1] O. C. Bridgeman, H. S. White and F. B. Gary, "Gasoline Vapor Pressures Permissible with 1931 Motors," *Proceedings*, Twelfth Annual Meeting, Am. Petroleum Inst., Section III, November, 1931, p. 40.

[2] O. C. Bridgeman, H. S. White and F. B. Gary, "The Present Status of the Vapor Lock Problem." *Proceedings*, Nat. Gasoline Assn. of Am., Table IV, p. 95 (1933).

It is shown graphically that 10 to 15 per cent of the cars on the road will vapor lock with 50 to 75 per cent of the gasolines in the summer time. In the spring, with unseasonably hot days, before all of the winter gasoline has been disposed of, vapor locking may be encountered more frequently by a somewhat larger percentage of cars. Most of the cars can tolerate considerably higher vapor pressures and some can tolerate extremely high vapor pressures, particularly in the cooler weather. *Only a few cars are responsible for the uneconomically low vapor pressures of present-day commercial gasolines.*

On the fuel side of the picture the A.S.T.M. committee's recommendations would outlaw many gasolines if the temperatures in the specifications were interpreted as "absolute maximum atmospheric temperatures." It is at once apparent from Fig. 2 how important it will be to gasoline marketers to have the temperature base for vapor lock specifications accurately and well defined so there will be no possibility of misinterpretation on the part of individuals or inspection agencies. It is certain that if "absolute maximum atmospheric temperature" were the basis, there would have to be a considerable change in the numerical limits to make the vapor lock specification agree with prevailing commercial practice.

Ainsley, Baker and Phillips have reported[1] a relationship between vapor locking fuel line temperatures and atmospheric temperatures for three cars with artificially heated fuel lines. Temperatures of the liquid in the fuel pump were determined by means of thermocouples. Because the fuel systems were artificially heated to produce vapor lock it is difficult to see how a valid relationship could be established between atmospheric temperature and vapor-locking fuel line (fuel pump) temperatures. Under the conditions of the experiments as described it would seem that any desired fuel line temperature could have been obtained at any atmospheric temperature. An additional contradiction is found in the fact that vapor locking at fuel pump temperatures below 90 F. is implied by the charts. This would require gasolines of at least 16 to 26 lb. vapor pressure. Yet it is stated, "in road test work commercial fuels purchased from service stations in the neighborhood were used." In the charts

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[1] W. G. Ainsley, L. E. Baker and E. B. Phillips, "Methods of Testing Automobiles for Potential Vapor Lock," *Proceedings*, Fourteenth Annual Meeting, Am. Petroleum Inst., Vol. 14, Section III, October, 1933, p. 22.

Mr. Brown presented in his paper, which deal with these data in more intimate detail, the highest Reid vapor pressure shown as used in these three cars was 14 lb.

One very pronounced discrepancy in the conclusions derived from the relationship is the use of fuel line temperatures during high-speed driving as the criterion for vapor pressure specifications to prevent vapor lock. Fuel line temperatures on idling and driving in city traffic were considerably higher. It would be interesting to know why these authors selected the driving condition which yielded the lowest fuel line temperatures. This is particularly interesting in view of Bridgeman's recent statement:

> "Due to the large vapor-handling capacity of many fuel systems, it was found impossible to get some of the cars to vapor lock on idling except with a fuel which would not permit operation at constant speed. Thus, although the fuel temperature increased on idling, the decrease in volume of fuel required more than compensated for the rise in temperature. Likewise, a number of cars would not vapor lock while accelerating after an idling period."

The fact that vapor locking fuel line temperatures as determined by the block tests should agree so well with road test vapor lock fuel line temperatures in three cars is a coincidence that would not be expected from the Bureau's recent work. This would mean that all four fuel systems had identical V/L ratios. It is further quite unexpected that the vapor locking fuel line temperatures should agree so well with the liquid "initial starting points." According to Bridgeman's recent work, vapor locking fuel line temperatures approach the liquid "initial starting point" (his predicted vapor locking temperature) only when the V/L ratio of the fuel system approaches zero and this condition was only found in cars with vacuum tanks. Each of the four fuel systems, however, had pumps instead of vacuum tanks.

It is with no intention of being destructively critical of what appears to be an excellent investigation that these comments are made. However, the conclusions to be derived from Bridgeman's recent work are at complete variance with the conclusions to be derived from the work by Ainsley, Baker and Phillips. There is inadequate definition somewhere. Before the two sets of data are used in conjunction, these differences should be determined.

From the viewpoint of petroleum economics it is utterly foolish to consider vapor pressure specifications as low as those proposed by the A.S.T.M. committee! The rapidity and universality with which the refining industry went to higher vapor pressures for winter gasolines should be strong enough evidence of the eagerness with which an over-abundant butane supply seeks the motor fuel markets.

Many of the objections of the automotive engineers to the proposed specification limits are based on the increasing speeds of new automobiles. It is contended that the investigations have not covered an adequate range of high speeds. This is quite true. On the other hand it is contended that not enough work has been done on the entire subject of vapor lock for an accurate conclusion. The work that has been done clearly demonstrates that comparatively inexpensive revision of new automobile fuel systems will accomplish such far reaching changes that such factors as car speed, driving conditions, atmospheric temperature and volatility of the gasoline become minor in the vapor lock problem.

On the subject of car speeds, very few of the cars which have been investigated are capable of sustained speeds much higher than the range studied. They either were not designed for such high speeds or they are now so old as to be incapable of high speeds or are unsafe at high speeds. From considerable driving experience it is a safe assertion that sustained car speeds of much over 60 miles per hour are not very imminent. Even on the prairies there are enough impediments to high speeds to make this an unlikely contingency for the immediate future. One of the greatest obstacles to speeds much higher than the average is not the speed itself but the inability of the other fellow to gage your progress. It is, no doubt, a safe observation that car speeds will rise slowly and universally rather than rapidly or sporadically. In considering sustained speeds of 80 miles per hour we are, therefore, contemplating for the most part cars to be built in the future. The petroleum industry is of the opinion that if such cars vapor lock on gasolines marketed under the proposed vapor pressure specifications or any inferences derived from these specifications, the reason will be the automobiles and not the gasolines. There are enough engineering data available to make vapor lock impossible and this can be done at practically no cost to the manufacturer of the automobiles.

There has been produced at great expense gasoline of high octane number to help make high top speeds possible. In so doing we now find that we have converted large quantities of our raw material into light hydrocarbons and that the increased volatility necessary to octane numbers has reduced the proportion of these light hydrocarbons that can be used in gasoline. Some far-seeing oil men anticipated this and have argued for rational design of fuel systems for the new cars of high top speeds. But now, in the year 1934, we are told that vapor locking is attributable to the very gasoline the automobile manufacturers have been asking for all these years.

Referring to Fig. 1 of Mr. Boyd's paper, I should like to call attention to the method by which vapor evolution was determined. The reference cited describes a method poorly controlled and regulated. It unnecessarily complicates the vapor lock problem to introduce another testing method. Mr. Boyd agrees that the Reid vapor pressure gives a useful indication of the vapor locking tendency.

Again with reference to Fig. 1 of Mr. Boyd's paper, what were the distillation characteristics of the four gasolines? Mr. Bridgeman's work on this variable demonstrated that considerable differences in distillation characteristics were necessary to attain substantial differences in practice. Mr. Boyd has subsequently furnished the following data:

	LOWEST (FIG. 1) 12-LB. GASOLINE	INTERMEDIATE (FIG. 1) 12-LB. GASOLINE	HIGHEST (FIG. 1) 12-LB. GASOLINE	14.5-LB. GASOLINE
Submerged initial boiling point, deg. Fahr.	113	...	108	102
Initial boiling point, deg. Fahr	90	86	90	90
4 per cent condensed, deg. Fahr	102	100	102	102
10 per cent condensed, deg. Fahr	115	113	108	116
30 per cent condensed, deg. Fahr	163	167	125	173
50 per cent condensed, deg. Fahr	219	223	144	217
70 per cent condensed, deg. Fahr	273	276	180	261
90 per cent condensed, deg. Fahr	341	343	255	334
End point, deg. Fahr	390	394	378	372
Recovery, per cent	97.5	97.3	98.0	94.7
Loss, per cent	1.5	1.5	1.0	4.3

The fact that two gasolines of similar distillation characteristics and identical Reid vapor pressure should display substantial differences in vapor evolution is worthy of note.

Under the heading "Relationship of the Proposed Schedule to Commercial Practice," the accuracy of all surveys in respect to Reid vapor pressures is open to serious question. It is common knowledge that survey samples are seldom if ever handled with enough care to assure representative samples for vapor pressure determinations. In our own surveys we attempt to be scrupulous in this matter, but to be as careful as complete accuracy requires would be too expensive.

Mr. Brown's observation is here decidedly applicable: "Any errors or carelessness in sampling tend to show a low vapor pressure."

In all the charts in both Mr. Boyd's and Mr. Brown's papers which correlate permissible Reid vapor pressure with either atmospheric temperature or fuel line temperature, Mr. Bridgeman's recent data fall in areas distinct from the results of other investigators and his own earlier work. As mentioned earlier, Mr. Bridgeman's recent work has demonstrated that there is a great difference between vapor locking vapor pressures and vapor locking fuel line temperatures as compared with permissible vapor pressures calculated from vaporization characteristics of the gasolines and non-vapor locking fuel line temperatures. Until this great difference has been accounted for it is not valid to compare any but tests conducted under actual and practical vapor locking conditions.

Mr. Boyd's Table I does not tell the whole story. Five cars might have been selected to show exactly the opposite trend. As a matter of fact, one popular low-priced car which never before has been a vapor-locker has a 1934 model which is susceptible to vapor lock.

There are no published data subsequent to 1932 but Mr. Bridgeman's surveys show the following average values for temperature rises above atmospheric at the pump inlet or vacuum tank inlet:

YEAR	STEADY DRIVING	IDLING
1930	29	43
1931	31	50
1932	34	48

This indicates retrogression rather than progress.

Automotive engineers seem to think an ideal fuel system is necessary. This is not the case. There are no ideal fuel systems, yet 70 to 85 per cent of the cars could tolerate high vapor pressure products under any circumstances. According to Bridgeman merely re-locating the fuel line will raise the permissible Reid vapor pressure 2 to $3\frac{1}{2}$ lb. All that is needed is fuel lines of adequate size located to avoid excessive heat input and sufficient vapor handling capacities to safeguard against vapor lock under extreme conditions, rarely encountered.

There is much to be said in behalf of Mr. Boyd's alternative suggestion. In the first place he provides a relatively greater factor of safety in cool weather than in warm weather. In the second place, there is much less possibility of misinterpreting his definition of atmospheric temperature. In the third place, his definition of Reid vapor pressure as in the fuel tank of the car is more closely related to the experimental work.

The designation of specific temperatures in accordance with Mr. Boyd's definition requires serious deliberation. It is reasonably certain that 95 F. is too low as a starting point. Commercial summer practice has demonstrated beyond serious question that 8-lb. gasolines have given reasonable freedom from vapor lock at atmospheric temperatures considerably above 95 F.

Mr. Boyd's suggestion is not far removed from the committee's recommendation. A compromise should be possible and the following is suggested:

TEMPERATURE OF AIR IN WHICH CAR IS DRIVEN, DEG. FAHR.	HIGHEST REID VAPOR PRESSURE OF GASOLINE IN FUEL TANK OF CAR GIVING FREEDOM FROM VAPOR LOCK IN MAJORITY OF CARS ON ROAD
100 and above	8
85	10
70	12

MR. NEIL MACCOULL[1] (*presented in written form*).—I wish to call attention to the fact that there is a distinct difference between boiling gasoline in the fuel system, and vapor lock. In some of the discussion on this subject these terms have been used interchangeably, as if they meant the same thing. I therefore like to divide this subject into two parts:

1. A gasoline will boil in passing through the fuel system of the car if it is raised to a temperature equal to its normal boiling point.
2. Vapor lock rarely occurs in cars except when the fuel temperature is raised considerably above its normal boiling point, because large quantities of fuel vapor can be separated from the gasoline before it reaches the fuel jet.

Most discussion of vapor lock indicates satisfaction if a gasoline can be used in a car without actually vapor locking. This, of course, is desirable, but I wish to call attention to the fact that a fuel should not be heated by the fuel system above its normal boiling point, even though vapor lock does not occur for the following reasons:

1. There will be an increase in the fuel consumption of the car due to the volume of vapor lost, which may be as great as 10 per cent of the fuel used by the engine.
2. The part of the gasoline lost as vapor represents the part which usually adds most to the anti-knock value of the gasoline.
3. If the car is allowed to cool off after the gasoline has boiled in the carburetor, starting when cold will be made more difficult, because the fraction lost by evaporation is the part which aids most in starting.
4. The loss of this gasoline vapor creates a fire hazard.

I am particularly pleased that Mr. Brown has called attention to the fact that any change in fuel specifications which will decrease the tendency to vapor lock will also increase the difficulty in starting a cold engine. Clearly, then, the only logical solution to this question is for engine and car manufacturers to reduce the temperatures of their fuel systems. By so doing, it may become possible satisfactorily to use gasoline that will start more readily at very low temperatures.

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[1] Director, Mechanical Dept., Beacon Research Laboratory, The Texas Company, Beacon, N. Y.

In tests we are conducting on 1934 cars, we have found that a steady run in an air temperature of 80 F. at 40 miles per hour up a 7-per-cent grade will raise the fuel system temperature to an average of 126 F. With the best car it was 119 F. and the worst car, 144 F. When idling after such a run, the temperatures rose appreciably for several minutes, the average for all cars being 146 F. The temperature of the worst car rose to 188 F.

It is my belief that, in this part of the country at least, vapor lock while idling at a signal light in congested traffic is much more frequent and annoying than the vapor which occurs at high running speeds.

Now it has been shown by Bridgeman and others that the normal boiling point of a gasoline (which is the temperature that would be recorded when distillation by the A.S.T.M. Method first starts, if the thermometer is submerged in the liquid) is approximately equal to the temperature for 10 per cent evaporated, as determined by the conventional A.S.T.M. Method.[1] It is therefore not a difficult matter to determine how high the 10-per-cent point of a gasoline should be in order to avoid boiling the fuel in these cars. It is also possible to specify this point in terms of Reid vapor pressure, since there is a definite relation between Reid vapor pressure and normal boiling point for any gasoline. For instance: 8, 10, and 12-lb. Reid vapor pressures correspond to normal boiling points of approximately 133, 119, and 108 F., respectively. Of course a distillation with the thermometer in the liquid is much simpler and more direct than obtaining a value from a Reid bomb.

In our experimental work these same cars were operated with gasolines of approximately $8\frac{1}{2}$ and $12\frac{1}{2}$ lb. Reid vapor pressure, the air temperature being raised until vapor lock occurred on idling after the standard run. With the $8\frac{1}{2}$-lb. gasoline, vapor locking occurred at an average temperature of 152 F., with a minimum of 141 F., although one of the cars did not vapor lock even when the fuel temperature had risen to 169 F. The normal boiling point of this gasoline was 139 F. With the $12\frac{1}{2}$-lb. gasoline, the average vapor locking temperature was 136 F., with a minimum of 110 F. The normal boiling point of this gasoline was 110 F.

It may seem strange at first glance that one of the cars vapor locked at the same temperature with each of these gasolines, and another car vapor locked with the $8\frac{1}{2}$-lb. gasoline but did not with the $12\frac{1}{2}$-lb. gasoline. The reason for this is explained in Fig. 1 of Mr. Boyd's paper which shows that while two gasolines may start to boil at the same temperature (and will thus have approximately the same Reid vapor pressure), one of them may give off a much greater volume of vapor than the other when they are both heated to any higher temperature. This is a very important fact, since a given car can handle only a limited volume of vapor before it will vapor lock.

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[1] Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products (D 86 – 30), 1933 Book of A.S.T.M. Standards, Part II, p. 778.

It is evident that the vapor locking tendency of a gasoline cannot be specified in so simple a manner as by a single Reid vapor pressure. It might be done with two Reid vapor pressures taken with different ratios of vapor to liquid in the bomb, or by the much simpler way of directly measuring the *liquid temperature* for two given volumes evaporated, such as 2 and 10 per cent.

But I wish to state again that the only logical solution to this whole question of vapor lock is to reduce fuel line temperatures so that the gasoline will not boil at any time. Under these conditions the only fuel data needed would be a measure of the normal boiling point, for which a single Reid vapor pressure would suffice.

MR. O. C. BRIDGEMAN.[1]—What puzzles me about the two papers presented is the fact that Mr. Boyd's data fall below the curve which seemed to give reasonable protection from vapor lock on the basis of the previous work. This lack of agreement, it seems to me, must be due either to differences in interpretation of what constitutes vapor lock or to differences in measuring equipment and technique. Unfortunately, Mr. Boyd has not gone into much detail regarding the instrumentation and technique employed in his work. Several questions arise as to the possible explanation of the differences.

In the first place, what is vapor lock? It has never been defined satisfactorily. It can possibly be defined as an interruption in the normal performance of the engine to an extent sufficient so that it is distinctly evident, but I do not know that one can go much farther than that. It is necessary to decide upon the criterion for vapor lock, and then to put the responsibility definitely on the operator and the observer as to whether vapor lock is occurring in accordance with the chosen criterion. This requires considerable training of the driver and observer, especially since they must not only have sufficient experience with each test car so as to know what the normal performance of each car is, but they must also be trained to overlook insignificant variations in performance. One possible explanation as to why Mr. Boyd's data differed from the previous data is that he is taking a criterion which differs from that taken in other work.

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[1] Physicist, National Bureau of Standards, Washington, D. C.

The temperature measurements are very difficult in the case of the gasoline flowing through the fuel system. The National Bureau of Standards had to do a lot of preliminary experimental work before an entirely satisfactory means of measuring these temperatures was developed. The temperature taken as the vapor-locking temperature is necessarily a minimum value, due to the fact that thermometers are located finite distances apart along the fuel system. The number of thermometers used and their location determine how much the observed vapor-locking temperature is lower than the actual vapor-locking temperature. In other words the permissible vapor pressure will be higher than that found by an extent dependent upon the instrumentation and installation.

The question of sampling is also important. The measured vapor pressure of the gasoline in the main tank will always be lower than the vapor pressure of the gasoline flowing through the fuel lines, the difference being dependent upon the method and care used in sampling. Even when sampling by water displacement and taking all conceivable precautions, errors as large as 0.5 lb. in vapor pressure may be obtained.

In concluding, we should not lose sight of the fact that it is necessary to compromise between ease of engine starting on the one hand and the occurrence of vapor lock on the other hand. If the vapor pressure of the fuel is made too low, engine starting may be difficult. If the vapor pressure is too high, extensive vapor lock difficulties may be encountered. Very little can be done in improving carburetor design so as to increase the ease of engine starting. On the other hand, many improvements can be made in fuel system design and installation which will keep more heat out of the fuel.

MR. GILBERT WAY.[1]—As a representative of one of the automotive companies, I should like to emphasize Mr. Boyd's statement wherein he says, "It is the obligation and desire of the automotive industry to make cars as free as possible from vapor lock."

You are all no doubt aware of the fact that a very considerable part of the work of an automotive engineering department is in dealing with service complaints from the field. It has been our experience that in instances of vapor lock difficulties the car is almost invariably blamed for the trouble. The average motorist rarely considers that the fuel might be of too high a vapor pressure, and in most cases he does not even know that vapor lock is occurring. Consequently it is absurd to think that an automotive manufacturer could ignore all complaints on the score of vapor lock and continue building a car with a poor fuel system. Mr. Boyd has shown that improvement has been made in lowering fuel line temperatures in the face of greater heat dissipation. It must be remembered that there are some very definite limitations in this respect due to existing induction systems, wherein it is universal practice to have the carburetor somewhere near the manifold and lead a fuel line to it from a pump bolted to the crankcase, all of which are necessarily under the same hood with a very hot engine and radiator, and an exhaust pipe that may be at red heat.

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[1] Chrysler Corp., Detroit, Mich.

Recent data obtained at our laboratory substantiate the statement that many cars now in use will vapor lock on gasolines of the proposed specifications at the atmospheric temperatures corresponding to them. Indications are that even the schedule of vapor pressures proposed by Mr. Boyd would cause trouble under conditions conducive to vapor locking, but by no means uncommon. I refer to the condition of starting after a short stop which has been made after a fairly high speed run. This is exactly what is encountered when traveling along the highway and a stop is made for some traffic conditions or re-fueling, and by no stretch of the imagination can this be called an exceptional or rare case. This condition while highly undesirable and annoying in level country operation assumes most serious proportions when applied to driving in hilly or mountainous regions. There the car will not clear itself up after a period of spitting and sputtering, as is usually the case on the level but will absolutely go dead on the grade, with the only alternative of backing off to the side of the road, which, to say the least, is a most uncomfortable situation and might even lead to disastrous consequences.

It is true that there are a great many owners of motor cars who drive very moderately and would never experience vapor lock, even on fuels exceeding the proposed specifications. However, if such cases were to influence our decisions on these matters, practically all progress would come to a standstill and no improvements would ever be made.

With all due respect to the work of the investigators on vapor lock, and there is no question but that all the data presented were carefully taken and are accurate, it is our belief that some of the data collected do fall under the category of the aforementioned "mild operation," and that the preponderance of such data on some of the curves is apt to be misleading.

The vapor pressures given in the latter section of Mr. Brown's paper of 11 lb. at 100 F., 14 lb. at 85 F. and 19 lb. at 65 F. for future use are beyond the scope of my imagination with my present limited experience with fuel systems.

There are sufficient data to show that the cars on the road at present will have trouble under certain frequently encountered conditions and that the proposed specifications do not offer as much protection as fuels with vapor pressures as now being marketed. We strongly feel that the alternative values as suggested by Mr. Boyd represent a much fairer compromise for the present. As to the future, it is our earnest desire to cooperate with the oil industries and we would, in addition to our own endeavors, welcome any suggestions that would help produce a foolproof fuel system that would enable the use of high vapor pressure gasolines without vapor lock or excessive vapor loss.

MR. G. G. OBERFELL.[1]—I should like to ask Mr. Boyd if the temperatures referred to in his Fig. 2 represent test temperatures? By this I mean, do the temperatures refer to tests taken of the atmospheric temperatures at the time the vapor lock tests were made? In other words, could you correctly apply to the curve shown the temperature taken outside of the car in the manner which Mr. Brown employed in his vapor lock experiments?

MR. T. A. BOYD.[2]—In our work atmospheric temperatures were measured by means of a mercury thermometer held at arm's length from the side of the moving car. This thermometer was shielded from the direct rays of the sun. The temperature thus obtained was then compared with the reading of a conventional Weather Bureau recording thermometer situated directly at the side of the track, and the two were found to be in agreement.

MR. OBERFELL.—Do you think they would correspond with results obtainable by Mr. Brown's method of determining atmospheric temperatures?

MR. BOYD.—There should be some correlation.

MR. OBERFELL.—I should like to have Mr. Bridgeman's opinion as to whether these atmospheric temperature test results agree with those obtainable by the method which he employed in his vapor lock experiments?

MR. BRIDGEMAN.—I should say driving on a concrete road that the temperatures would be higher than would be those reported by the Weather Bureau.

MR. OBERFELL.—Apparently Mr. Boyd is almost in agreement with the committee's recommendations; the difference between his recommendation and those of the committee amounts to about 1 lb. or less. Considering the errors which may be encountered in experiments of this kind, it is my opinion that the recommendations are in as close agreement as could be expected. At least this much discrepancy was to be anticipated between the two recommendations, especially since Mr. Boyd has been so careful in choosing the safest conditions one would expect to consider so far as reasonable freedom from vapor lock is concerned.

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[1] Vice-President in Charge of Research, Phillips Petroleum Co., Bartlesville, Okla.

[2] Head, Fuel Section, Research Division, General Motors Corp., Detroit, Mich.

There has been much criticism about the use of the expression "atmospheric temperature." I do not think this criticism is justified. The committee members certainly knew they were referring to "test temperatures" whenever the expression "atmospheric temperature" was employed. The committee had no intention of attempting a correlation of test temperatures with climatic conditions. There would have to be a tremendous amount of data compiled from the U. S. Weather Bureau records if such a correlation were attempted. I think this latter problem has more to do with shipping schedules than it has to do with motor fuel specifications. No one would consider it necessary to have a motor fuel specification that would assure freedom from vapor lock at the maximum atmospheric temperature ever recorded. It is not necessary to have a specification that would assure freedom from vapor lock at an atmospheric temperature that would be encountered only once in a hundred years and only at a time when one would expect to drive his car at a speed of 80 miles per hour. To provide for protection for freedom from vapor lock for all cars at the maximum atmospheric temperature ever recorded by the U. S. Weather Bureau is not within the bounds of practical motor fuel specifications.

Much criticism has also been directed against the use of the Reid vapor pressure as a specification for insuring reasonable freedom from vapor lock. We are not interested in showing a relation between the Reid vapor pressure and the vapor lock temperature. The Reid vapor pressure test is intended to provide a test condition that will evaluate the worst conditions to be encountered and thereby insure a means of determining a test value that will serve as a guide for the protection of the majority of cars. The Reid vapor pressure testing method embodies principles that make it an ideal one for this purpose. In the present Reid vapor pressure testing method the volume of the air bomb is four times that of the liquid bomb. The vapor handling capacities of most of the cars in the majority of instances are greater than this ratio. In this connection I want to emphasize that there are two problems to be considered when contemplating a vapor pressure specification as a means of safeguarding from vapor lock difficulties. One is that the numerical value should be so low that protection will be provided for the majority of the cars. The other problem resides in the fact that the method of determining the vapor pressure should be such that it will represent the worst conditions encountered in actual practice. Both of these principles are incorporated in the motor fuel specifications recommended by the committee. It has put a rigid interpretation on the method of taking the test and a rigid interpretation on the actual values determined by that method. In my opinion the recommendations of the committee are highly conservative in this respect.

I should like to call attention to the fact that Reid vapor pressures much higher than those recommended by the committee can be used in many instances. In criticizing the Reid vapor pressure testing method Mr. Boyd showed that different gasolines of the same Reid vapor pressure gave off different amounts of gas when heated to the same temperatures above 100 F. This argument carried to extreme would be represented by a constant-boiling compound such as pentane and in this connection I should like to refer to some road tests conducted with commercial pentane which had an end point of 97 F. and a Reid vapor pressure of 17.5 lb. This product was driven in a Chrysler Imperial 8 at a speed of 60 miles per hour when the atmospheric temperature was 60 F. No trouble from vapor locking was encountered either while driving or upon subsequent idling.

MR. W. A. SMITH.[1]—We have had quite a little vapor lock difficulty on stop lights under heavy traffic conditions this year, and in analyzing the reason therefor we came to the conclusion that we could not place much confidence in the Reid vapor pressure as an absolute test. As a suggestion to the committee I should like to give out this one thought: In 1931 we were selling a 50 per cent distilled gasoline at 270 F. In 1932 that had dropped to about 250 F. In 1933 it had gone down to 235 F., and in 1934 it was about 210 F. In 1931 we were selling a 9-lb. gasoline, in 1932, 8½-lb., in 1933, 8½-lb., and we had to drop it to 8 lb. this year. There must be some reason for that. I think Mr. Boyd's paper gives the answer. The larger amounts of low boiling fractions are responsible for the difficulty.

MR. G. G. BROWN.[2]—In view of the discussion it is clear that there are different points of view as to the object to be accomplished by recommending specifications to insure reasonable freedom from vapor lock so far as the fuel is concerned. I believe that every member of the Vapor Lock Section thoroughly appreciates that the recommended limits will not prevent vapor lock on all cars under all conditions. In order to do so would demand the use of fuels of lower vapor pressure than are now in common use, as vapor lock is now being experienced on many cars under severe conditions throughout the country. If an attempt were made to so limit the vapor pressure of fuels as absolutely to prevent vapor lock on all cars under all conditions of service, it would be found that starting would become extremely difficult, if not impossible, in many parts of the country at times when the temperature varies over wide limits within a few hours or few days.

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[1] Manager, Gasoline and Oil Depts., Larkin Co., Inc., Buffalo, N. Y.

[2] Professor of Chemical Engineering, University of Michigan, Ann Arbor, Mich.

For this reason, leaving out of consideration the relative responsibility of the car and the fuel for causing vapor lock difficulty, and considering solely the user from the standpoint of ease of starting on a cold morning and freedom from vapor lock when using the same gasoline a few days later when the temperature may be much higher, it is clear that the setting of limits to insure *reasonable* freedom from vapor lock so far as the fuel is concerned is a *compromise.*

When in addition to these factors, it is considered that the light hydrocarbons which may be included in greater quantity in gasolines of higher vapor pressure possess extremely high anti-knock property, and must be wasted if not included in motor fuel, the economic importance of reaching a proper compromise may be appreciated. By relatively simply modifications in the fuel system, practically all motor cars now experiencing difficulty with vapor lock may be made to handle fuels of the recommended vapor pressure without difficulty. This statement is supported by the data reported by Boyd in his Fig. 2, where it is indicated that if the fuel systems of the worst cars are modified so as to lower the maximum temperature of the gasoline about 8 deg. Fahr., vapor lock would be entirely eliminated with fuels of the recommended limits, when such fuels are used under severe driving conditions at a temperature as recorded by a Weather Bureau station equal to the maximum for the allowed vapor pressure.

I should like again to emphasize the fact that relationships such as reported in Fig. 3 of Mr. Boyd's paper which gives the Reid vapor pressure of the gasoline and the observed vapor locking temperature of the fuel are apt to be most unreliable unless extreme care is taken to determine the observed vapor locking temperature only under vapor locking conditions and at the exact point at which vapor lock occurs. Since this is difficult, the committee has taken the position that actual data such as reported in Fig. 2 of Mr. Boyd's paper are far more reliable for purposes of recommending vapor pressure limits.

These recommended limits will insure reasonable protection from vapor lock so far as the fuel is concerned, because many cars are free from vapor lock with such fuels even under the most severe conditions, and those now experiencing difficulty can be brought into line without difficulty. Therefore a reasonable compromise demands that the car manufacturers so modify their fuel systems in those particular cases where vapor lock is now experienced as to eliminate that difficulty.

MR. BOYD.—The distillation characteristics of the three 12-lb. gasolines and the one 14½-lb. gasoline requested by Mr. Alden were furnished to him as set forth in his discussion. Also I shall be glad to supply any one interested with information about the form of apparatus and procedure used in determining vapor evolutions. This direct method of determining the vapor-evolving characteristics of a gasoline is based upon the work of Francis.[1] The actual procedure and apparatus used in getting the data on the volumes of vapor evolved, as presented in Fig. 1 of the paper, were developed by M. J. Mulligan of our staff. In this method the volumes of vapor evolved at the various significant temperatures were measured directly over dry mercury. The results obtained were definite and reproducible.

The answer to the question about why, on gasolines of given Reid vapor pressures, the results obtained in our work show vapor lock as having occurred at temperatures considerably lower than those at which vapor lock was observed in the cited work of Bridgeman is simply that the 40 mile per hour operating conditions under which Bridgeman's tests were run were altogether too mild to be representative of service conditions. Drivers customarily run 60 miles per hour and more, and that was therefore done in our work.

With regard to the kinds of disturbances which were called vapor lock in our work, it may be said that they were not in any sense questionable types. As is indicated in Fig. 2 of the paper, they consisted of the following unmistakable forms: engine would not start after a brief stop, engine would not idle, engine failed completely during acceleration after standing, car would not run at high road speeds on the level or it failed completely at wide-open throttle on the hill.

As regards method of sampling, it may be noted that in our work the samples of gasoline used in getting the data presented were taken from the fuel tank of the car by withdrawal through the drain plug in the bottom of the tank and collected by water displacement in a completely closed system. Following collection, the best known technique was employed in handling the samples and in measuring the Reid vapor pressure of each. Careful measurements were also made to determine how the Reid vapor pressure of the gasoline taken from the fuel tank of a car compared with that in the filling station tank from which it was filled. It was found that the two values agreed within one or two tenths of a pound, or well within the experimental error of the Reid vapor pressure determination.

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[1] C. K. Francis, "Method for Determining Vapor Lock," *Oil and Gas Journal*, March 17, 1932, p. 22.

BITUMINOUS JOINT COMPOUNDS FOR SEWER PIPE

By J. S. Crandell[1]

Synopsis

Leakage into or from sewers occurs almost always at the joints. To make joints that are tight and that will remain so during the life of the sewer requires a material that is proof against water, acids, alkalies, gasoline, motor oils, heat, shocks, and settlement of the pipe. It is also desirable that it be durable, inexpensive, easy to handle on the job, repellent to roots of trees and shrubs, and somewhat pliable.

This investigation includes a study of some of the physical properties of a score of bituminous joint compounds for sewer pipe now on the market, together with others that have not yet been manufactured commercially. The tests were made using 6-in. vitrified clay sewer pipe, and were performed in the Materials Testing Laboratory of the University of Illinois under a Fellowship agreement with the Clay Products Association of Chicago. Tests were made of the ability of the filled joints to remain water-tight when subjected to internal and external water pressure; the resistance of the joints to flexure; the solvent action of light petroleum oils; the action of sewage on the jointing materials; the adhesive qualities of the compounds; the determination of the rate of settlement of mineral fillers in bitumens; the comparison of cold plastic materials with hot-poured types; and the evolution of several new compounds containing bitumen and rubber.

All but one of the compounds produced joints able to withstand pressure. If mineral fillers are added to bitumen they will settle to some extent, especially during the period of heating. Gasoline in sewage has a solvent effect on asphalts, but coal-tar pitches are unharmed. The addition of mineral fillers to pure asphalts will reduce their tendency to foam when hot and if brought in contact with water. Satisfactory cold plastic materials were produced from powdered asphalt. Ordinary house sewage had no effect on any of the compounds. Adhesion tests were unsatisfactory. Rubber suspensions in asphalt produced new materials, one of which apparently is very satisfactory.

Pressure Tests:

In making the pressure test, the joint was prepared by blocking the position of the pipes, calking with jute, and placing a rubber hose runner in front of the bell. The bitumen was heated to the proper temperature and immediately poured into the joint. It was then allowed to cool to room temperature.

A plumbing test plug was placed at each end of the pipe which was then filled with water, and pressure applied. In practice, a sewer pipe is not ordinarily subjected to a pressure of more than 5 lb. per sq. in., although there may be occasions where greater pressure may be exerted for short periods. In this test the pressure was increased in 5-lb. increments to a maximum of 30 lb. All of the materials tested were satisfactory at 5 lb., and but one of them failed at 30 lb. However, the test brought out the following other features of practical interest.

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[1] Professor of Highway Engineering, University of Illinois, Urbana, Ill.

Tiny cracks and imperfections in tile pipe that are not patent to the naked eye often cause leakage around the joint. The joint compound appears to have failed, whereas, in reality, it is perfect. Hence, if the pipe is to be under pressure, each individual tile should be most carefully handled and inspected before laying. Again, wet joints may cause the asphaltic compound to foam so excessively that the joint cannot be filled. This is true of all pure asphalts. If a mineral filler is mixed with the asphalt, excessive foaming may be prevented. The percentage of filler varies with the asphalt used, but apparently it should be between 30 and 50. If the amount of filler is too great, the compound is so stiff that it will not penetrate to the bottom of the joint. Excessive foaming causes bubbles to form on the pipe, causing the joint to be materially weakened.

And finally, it was observed that in those compounds made with mineral fillers and bitumen, there is a marked tendency toward separation of the ingredients. This led to the next tests, the settling tests.

Settling Tests:

While the investigation was in progress it had been noted that the samples containing mineral fillers had shown a difference in quality and texture at the top and the bottom of the containers. It seemed advisable to ascertain how much settling occurs when the compound is packaged, and how much settles out when the compound is heated in a kettle on the job.

To test for the former, samples were taken from the top and the bottom of the containers on their arrival and at regular periods thereafter. To test for the latter, a funnel with an interior angle of 60 deg. was made; the sides were steep enough so as to be above the angle of repose, even for a very plastic material. A removable cup was placed at the bottom to catch the settled filler. The material under test was taken from fresh factory shipments and placed in the funnel which was then kept in an electric oven for 3 hr. After cooling, the cup was removed and samples were secured from it. At the same time, samples were taken from the material at the top of the funnel. Analyses were made.

Apparently all mineral fillers tested settle. The settlement takes place not only in the heating kettles but also in the containers shipped from the factory. Those fillers whose specific gravities are close to that of the bitumen settle more slowly, and to a lesser degree than the heavier ones, but they *do* settle. The settlement might prove to be objectionable if the containers are large and if the compound contains as much as 45 per cent filler, by weight.

As soon as even a slight concentration of filler occurs at the bottom of the kettle, the circulation of bitumen is retarded, and very soon over-heating begins which develops into coking. The percentage of filler that will make the compound too stiff to flow varies with each combination of filler and asphalt. However, this point is around 50 per cent by weight. This fact is of importance to the man in the field, because, when this "fluid limit" is reached or exceeded, the compound begins to coke immediately. Burned out kettles are the result. Constant stirring in the kettle is necessary with such materials.

Resistance to Solvents Test:

Although most city ordinances prohibit the dumping of gasoline and oils into sewers, it is the practice of filling stations and garages to disregard the law. A considerable amount of these materials will be found in the average sewer. Tests were run to determine whether such materials are harmful to bituminous joint compounds.

Observation of the solvent action of petroleum ether on the bituminous compounds in actual joints was made. Two hundred milliliters of petroleum ether were put in a pipe, the ends of which were plugged, and the jointing compound was watched twice a day until failure occurred with the asphaltic compounds. The ether had no apparent effect on the coal-tar pitch compounds. The asphalt failed by oozing from the joints on the fourth day. It will be noted that there was nothing in the pipe but petroleum ether, and hence the compounds were exposed to this solvent in concentrated form. In real sewage, the percentage of gasoline to water is trifling, and hence there is probably little solvent action actually taking place. Furthermore, if the solvent action is great enough to soften the compound to any extent, the pressure of the surrounding earth will hold the compound in place until the excess solvent has volatilized.

Another test was made by placing samples of similar size in flasks containing 25 ml. of petroleum ether and noting the rate at which the samples were dissolved. There was little difference among the pure asphalts, but the compounds with fillers showed great variation. The rate of dissolving decreased directly with the increase in the amount of filler. The solvent action on the coal-tar compounds was negligible.

It seems from these tests that the gasoline and oils present in sewage have little solvent action on bituminous compounds.

Stability Tests:

Jointing compound should stay in place for the life of the sewer. It therefore should not disintegrate, flow, nor creep. In order to ascertain how the compounds would act, from a laboratory standpoint, joints were filled and the pipes were placed on racks in a room kept at 24 C. (75 F.). This was a rather severe test since there was no earth about the joints to help hold the compound in place.

It was found that those asphaltic compounds with high penetration would flow soon after the form that held them during setting time was removed. If these high-penetration asphalts were stabilized with filler, they do not flow.

The coal-tar pitch jointing compounds used in this work were evidently not suitable, since even the very hard pitches flowed at room temperature. However, at a later date, and when the investigation was almost over, a pitch compound was submitted which has not flowed after a period of 6 months. It seems to be the most stable of the coal-tar products examined.

Adhesion Tests:

Time and energy were expended to try to find a good, reliable, satisfactory adhesion test. The results to date are zero.

New Compounds:

In some types of field work it is handy to use a jointing material that can be applied cold, much as plaster is used. Numerous materials were experimented with, but all of the early attempts ended in partial failures. Eventually a material was produced that seems to be satisfactory. It is made by mixing an asphalt cement of high penetration with powdered asphalt dissolved in benzol. The product is a plastic compound that may be either troweled into the joint, or shaped to form and pressed into place with a jack. The first method would be excellent for patch work, and the latter would be of use on big jobs where speed of construction is desirable. This compound would have to be used with some degree of speed and skill since the benzol evaporates rapidly. After the joints have been filled, they should stand exposed for 5 hr. or more, and the sewer system should not flow to its full capacity for a week, as pressure from within may cause the partially-set compound to become dislodged. The joint can stand external pressure up to 30 lb. per sq. in. immediately if first calked with jute and then well filled with the plastic compound. This material is resistant to solvents.

Rubber-Asphalt Compounds:

It has always been difficult to fill successfully the joints of a vitrified clay sewer pipe when the joints are partially full of trench water. And it has been considered impossible if the pipe is submerged. As previously noted, foaming occurs unless the compound is well loaded with filler, in which case the compound may be so stiff that it stops flowing when it strikes the water in the trench. Mr. F. R. Grant conceived the idea that a mixture of rubber and asphalt might solve the problem. After many experiments he produced a compound that has several unique features. Apparently it is a true colloidal suspension.[1] The compound pours readily at 185 C. (365 F.), and is not harmed by being kept at a temperature of 225 C. (435 F.) for 2 hr. It may be poured into a joint submerged in water, and will fill it without excessive foaming. It is not brittle at 0 C. (32 F.), and it is stable at 100 C. (212 F.). It is attacked by solvents to the same degree as pure asphalt.

This new material has so many advantages over some of the older types that it will be well to watch its development. Of course, sufficient time has not elapsed since its invention to show whether it has long-time lasting qualities.

Thirty-three compounds were tested. Each was given a number, and throughout the tests the materials were known only by number. Laboratory tests were run and data recorded on all of the compounds that were used. These tests agreed closely with those run by the manufacturers.

The information presented in this paper is a part of that contained in a manuscript in preparation for a *Bulletin* of the Experiment Station of the University of Illinois.

SUMMARY

1. All of the materials tested withstand internal and external water-pressure such as is encountered in ordinary sewers.

2. Vitrified clay pipe that has been broken, to even the slightest extent, should not be used.

3. Asphaltic materials containing admixtures of fillers (40 to 50 per cent, by weight) can be successfully poured when water is present in the trench, but pure asphalt, or asphalts with a low percentage of filler will foam so excessively as to make an imperfectly filled joint.

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[1] Patent applied for.

4. All poured jointing compounds tested containing fillers in amounts over 10 per cent show a marked tendency for the fillers to settle and thus cause the material to overheat, coke, and burn.

5. Gasoline, petroleum ether, and other similar solvents found in sewage will destroy asphalt compounds if exposed for a long time, such as two or three days, but, in practice, there is no harm done if the solvents are washed away fairly rapidly.

6. Coal-tar pitches are not affected by the ingredients in sewage, but they have a tendency, except in one instance, to flow out of the joint.

7. A cold plastic compound can be made from powdered asphalt, a suitable flux, and a solvent. This compound has all the requirements of a good jointing compound.

8. A poured compound of rubber and asphalt was produced that was successful.

DISCUSSION

Mr. H. B. Pullar[1] (*by letter*).—It was my pleasure some time ago to witness tests at the University of Illinois on the rubber asphalt compound referred to in Mr. Crandell's paper. I was agreeably surprised at the results which this asphalt-rubber mixture seemed to give. While it should still be placed in an experimental classification, its potential possibilities are most excellent, and the results already accomplished fully warrant and justify further study of this type of material.

In my experiences I have found that certain types of blown asphalts are most suitable for joint compounds where the sewer pipe is dry and where the pure asphalts can be properly applied; but they have proved very unsatisfactory in wet joints or when applied under abnormal conditions. The asphaltic compounds, usually made by mixing silica sand or dust with pure asphalt, have overcome to some extent the deficiencies of the pure materials. However, these compounds, as stated in the paper, tend to separate when heated and require more skill and attention for proper handling than the average conditions permit.

If it is possible to combine the attractive uniform qualities of the pure asphalt and the adhesive and pouring properties of the asphalt compounds into one material, it will be most desirable.

The successful experiments conducted with the rubber asphalt compounds would indicate that they might be the answer to a satisfactory bituminous jointing compound.

Mr. C. S. Reeve.[2]—I have strongly questioned the peculiar behavior of one of the samples of coal-tar pitch material referred to in the paper. There is nothing in the data or in the tests of the physical properties of this material to show just why it behaved as peculiarly as it did. We can see no reason why a pitch compound, as has been shown since, should not be prepared that will meet the requirements that it is expected to meet.

So far as the leakage goes, and this applies I believe to a great many samples, that is due, as Mr. Crandell has said, not to the compound itself but to defects or slight imperfections in the clay pipe. As I gather, practically all of these materials are applied without a primer. I wonder whether a great deal of the trouble could not be overcome if a priming coat were used before the application of the compound.

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[1] General Manager, American Mexican Petroleum Corp., Chicago, Ill.

[2] Manager, Research Development, The Barrett Co., Leonia, N. J.

I understand that the information secured is to be used during the coming year in the formulation of specifications. Some specifications have come to my attention recently in which there has evidently been an attempt to correlate the stability of the material with some simple form of test. One in particular is a small cylindrical specimen—roughly, I should say $\frac{3}{4}$ in. in diameter by 2 in. in length, subjected to, I think 140 F. (60 C.) for 2 or 3 hr. without any appreciable slump. Some of the compounds stand that test very well, and I should be interested in knowing if Mr. Crandell has had any experience with that particular slump test, and whether he has any correlations of that test with the stability test which he has made.

There is another test which is a modification of the one described in this paper that might be considered, and that is a settling test developed by the Pennsylvania State Highway Commission. A rather complicated apparatus has been developed for determining settlement in joint fillers, but while it is somewhat complicated, it comes pretty close to putting that test on a more or less standard and reproducible basis.

MR. J. S. CRANDELL.[1]—Mr. Reeve has referred to the use of primers before the application of the compounds. We used the compounds exactly as the manufacturer directed.

MR. J. S. MILLER, JR.[2]—Having been interested for some twenty years in asphalts carrying mineral fillers as well as those in which mineral filler has been incorporated, it comes as somewhat of a surprise to hear that a mineral-filled product shows settlement of the filler when such products are stored at temperatures of 77 F. (25 C.). I should like to ask Mr. Crandell if the filler in the material in question had any peculiar properties or was its particle size larger than what is generally termed filler or was the continuous phase of the product a liquid material such as a cut-back? Can he in any way account for the apparent stratification that was found in this instance?

MR. CRANDELL.—All fillers settle so long as there is more than 10 per cent of bitumen present. It does not matter whether the bitumen is solid, semi-solid, or liquid, the fillers settle. That was what impressed us. If a sample is taken from the top, from the middle, and from the bottom of the container, the three are totally different. We did not believe it at first ourselves, but after going through this test for four years we came to the conclusion that it was as stated.

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[1] Professor of Highway Engineering, University of Illinois, Urbana, Ill.

[2] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

MR. MILLER.—Were the samples from the top and bottom of the material taken when the material first arrived? May not the segregation have occurred when the packages were filled initially? In the case of poorly chosen fillers and where the filled asphalt is drawn at too high a temperature, the condition described by Mr. Crandell is very apt to happen, hence our interest in knowing whether the material was stratified upon receipt and whether it continued to change during storage.

MR. CRANDELL.—We took the samples as soon as the material was received from the manufacturer. We found sometimes that if the Pennsylvania compounds were in the laboratory for a week there was a settlement as early as that. Samples taken later showed further settlement, although kept at room temperature.

MR. M. H. ULMAN.[1]—We have used for a number of years in Pennsylvania with excellent results a bituminous material impregnated with mineral flour for sealing of joints and maintenance of cracks in pavements. The so-called "settling test" to which Mr. Reeve referred, was developed in order to obtain a material in which excess settling does not occur when stored or when maintained at a temperature of 325 F. (163 C.) for a specified period of time.

I am therefore unable to reconcile the amount of settling which Mr. Crandell stated he had obtained in his experiments on material prepared under the Pennsylvania specifications. I would naturally wonder if the material on which his experiments were based complied with requirements of our settling test. In a typical analysis of material obtained under our Specifications, J-1, there was found 23.2 per cent of mineral flour insoluble in carbon disulfide. After maintaining this material in our settling tests for a period of two hours at a temperature of 325 F. (163 C.) the material from near the top analyzed 23.2 per cent insoluble in carbon disulfide and from the bottom of the container 24.9 insoluble in carbon disulfide. This example indicates the small amount of settling in the material obtained under our specifications when maintained in a heated condition.

It is essential to use mineral flour of the proper degree of fineness to minimize the amount of settling.

MR. W. H. HERSCHEL.[2]—It is entirely in accordance with theory, that if you have a solid suspended in a liquid, it makes no difference how highly viscous that liquid may be, if you give it time enough, the solid will settle out of that liquid. I should like to ask Mr. Crandell whether in all cases the fillers that settled were solids?

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[1] Assistant Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

[2] Associate Physicist, National Bureau of Standards, Washington, D. C.

MR. CRANDELL.—Yes, they were. We determined the specific gravity of the fillers and found that it had something to do with the time of settling. Thus, the lighter ones took much longer. Some of the heavy ones would settle in the course of 3 months, so that there was a layer at the bottom. That is one of the reasons why our men in the field are having such difficulty. When heated in kettles it cokes before you know it. The material should be constantly stirred; but this is not done unless the inspector is unusually alert.

MR. E. C. ISOM.[1]—I should like to ask Mr. Crandell whether any work has been done to combat the solvent action of petroleum hydrocarbons or similar materials which have a deleterious effect on bituminous joint compositions, by the admixture or the use of small amounts in varying proportions of synthetic resins in such compositions. In some of our laboratories we have done a little experimental work in the combination of various types of synthetic resins with bituminous products, and while we have not developed the matter to any great extent we found some very interesting results in increasing the resistance of these compounds to the solvent action of hydrocarbon oils. Of course, we are more interested in that field from the pipe-line standpoint, because a leak in a transcontinental pipe line coated with a bituminous composition immediately destroys the pipe coating. It occurred to me that if the solvent problem in any certain locality is very great, something might be done to correct it along these lines.

MR. CRANDELL.—I know of no such work that has been done to date.

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[1] Manager, Paint Dept., Sinclair Refining Co., New York City.

INDENTATION OF ASPHALT TILE[1]

By J. W. McBurney[2]

Synopsis

This paper presents a formula for relating the depth of indentation to the time during which a loaded sphere acts upon such a plastic body as an asphalt tile. Data are presented showing the effect of varying the diameter of the sphere and the load. A portable instrument for indentation testing is described. The use of these test methods in technical specifications for asphalt tile is discussed.

Introduction

The material, asphalt tile, considered in this paper, is defined[3] as follows:

> "Asphalt tile shall be composed of a thoroughly blended composition of thermo-plastic binder of the asphaltic and/or resinous type, asbestos and/or raw cotton fibers, and inert materials as fillers or pigments, formed under pressure while hot and cut to size."

The testing methods here reported were developed as part of an investigation of the properties of asphalt tile, preliminary to the writing of a technical specification for asphalt tile as a flooring material. When this investigation was begun (1932) each manufacturer had a method of testing for indentation peculiar to his own plant. At various plants, various loads acted on various shapes and sizes of indenting tools for various times at various temperatures. The only indentation test recognized by the industry as a whole was that prescribed by the Veterans' Administration Specification in which the Riehle hardness tester for soft materials is used with a load of 120 lb. acting for 30 sec. at 77 F. (25 C.) through $\frac{3}{4}$-in. spheres. In 1932 this specification called for an indentation of not less than 0.014 in. or more than 0.022 in. under the method of test specified.

It was becoming increasingly evident that this specification did not control indentation behavior in service. Some tile purchased under this specification indented badly, and others under apparently similar conditions gave complete satisfaction. Cases were met where tile much harder than that permitted by the limits 0.014 to 0.022-in.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Research Associate of the Asphalt and Mastic Tile Assn. at the National Bureau of Standards, Washington, D. C.

[3] Proposed Federal Specification for Asphalt Tile, December 7, 1933.

indentation failed by excessive indentation in service. Obviously the depth to which a ball penetrated at a single temperature when acted upon by a single stated load for a period of 30 sec. did not give enough information concerning the behavior of an asphalt tile. This paper presents the results of attempts to learn more about the behavior of asphalt tile under load.

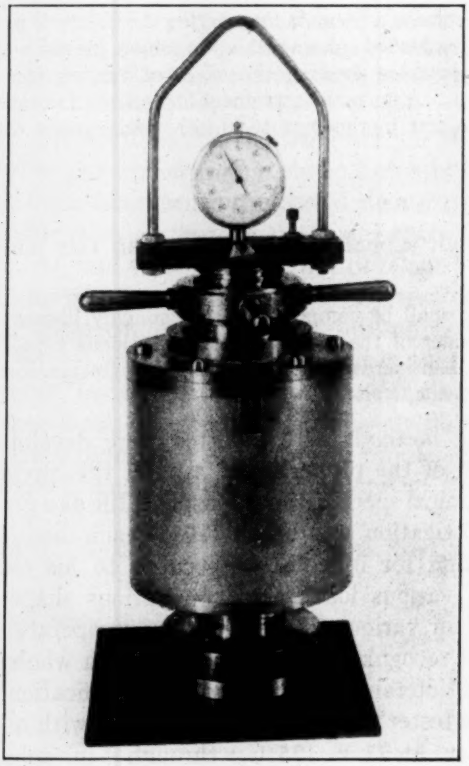

Fig. 1.—Portable Indentation Tester for Asphalt Tile. The test specimen shown is 6 by 6 by $\frac{3}{16}$ in. in size.

Test Apparatus

The apparatus used in much of this work is shown in Fig. 1. By it a sphere $\frac{1}{4}$ in. in diameter, and carrying a load of 2 lb. is lowered until it is in contact with the surface of the test piece. The dial micrometer is then set to zero. An additional load of 28 lb. is then added by suitably rotating the nut. The resulting depth of indentation is read from the dial micrometer at appropriate time intervals. The special features of this particular apparatus are: first, it is easily

TABLE I.—INDENTATION TESTS ON SAMPLES OF ASPHALT TILE AT 77 F. (25 C.), 30-LB. LOAD ACTING ON ¼-IN. DIAMETER BALL (ALL READINGS IN MILS).

Test	Observed Reading at 1 min.	Observed Reading at 10 min.	Observed Reading at 24 hr.	24-hr. Reading Calculated from 1 and 10-min. Readings	Change in 1-min. Reading to Make 24-hr. Calculated Check with Observed 24-hr. Readings	Percentage Deviation of Calculated 24-hr. from Observed 24-hr. Readings
No. 1	12.2	16.8	31.8	33.5	+0.3	+5.3
No. 2	11.2	16.5	39.2	38.1	−0.2	−2.8
No. 3	11.1	16.0	37.2	35.2	−0.2	−5.4
No. 4	11.8	16.0	28.8	30.9	+0.4	+7.3
No. 5	12.3	16.5	30.3	31.1	+0.2	+2.6
No. 6	10.8	15.5	31.8	33.8	+0.3	+6.3
No. 7	12.0	15.8	29.5	28.0	−0.2	−5.1
No. 8	9.8	13.5	27.0	27.0	0.0	0.0
No. 9	10.4	14.7	28.2	31.0	+0.5	+9.0
No. 10	9.8	13.2	23.8	25.1	+0.3	+5.5
No. 11	11.0	14.9	27.0	28.7	+0.3	+5.9
No. 12	11.0	15.8	33.8	34.5	+0.1	+2.1
No. 13	10.6	15.2	33.5	33.1	−0.1	−1.9
No. 14	11.0	16.0	36.0	35.9	0.0	−0.3
No. 15	13.3	19.0	41.8	41.0	−0.1	−1.9
No. 16	11.2	16.0	33.5	34.6	+0.2	+3.3
No. 17	12.5	17.3	34.0	34.9	+0.2	+2.6
No. 18	14.3	19.9	38.7	40.6	+0.3	+4.9
No. 19	11.1	15.2	30.0	30.0	0.0	0.0
No. 20	12.5	17.3	34.0	34.9	+0.2	+2.7

TABLE II.—EFFECT OF VARYING LOAD AND DIAMETER OF SPHERE ON INDENTATION AND AVERAGE PRESSURE OF A PARTICULAR SAMPLE OF ASPHALT TILE AT 77 F. (25 C.).

Diameter of Sphere, in.	Load, w, lb.	Indentation, mils		m	a	b	Calculated Time and Indentation Extrapolated to $di/dt = 0.001$[a]		Corresponding Average Pressure, lb. per sq. in.	
		1 min.	10 min.				Time, sec.	Indentation, mils	Load, spherical area	Load, paraboloidal area
1	120	17.0	22.5	0.1218	10.3	0.086	3 380	27.8	1375	1340
1	90	14.5	19.1	0.1196	8.9	0.099	2 740	22.9	1250	1225
1	60	10.3	14.8	0.1575	5.4	0.090	3 000	19.1	1000	980
1	30	5.0	7.5	0.1761	2.4	0.080	1 560	8.9	1070	1065
½	120	27.9	37.0	0.1226	16.9	0.141	6 020	49.1	1560	1425
½	90	20.5	28.2	0.1384	11.6	0.129	5 270	38.1	1510	1400
½	60	15.0	21.0	0.1461	8.25	0.138	4 050	27.4	1395	1325
½	30	7.9	12.0	0.1816	3.75	0.125	2 900	16.0	1195	1155
¼	120	36.0	50.1	0.1435	20.0	0.167	10 900	86.0	1785	1380
¼	90	30.2	41.1	0.1338	17.5	0.195	7 740	62.4	1844	1505
¼	60	22.4	31.0	0.1412	12.6	0.210	5 950	43.2	1775	1530
¼	30	12.7	18.4	0.1610	6.5	0.217	4 020	25.0	1555	1400

[a] This is a rate of 0.000001 in. per sec.

portable, weighing 42 lb.; second, readings can be made on floors in place, since the instrument is designed to be supported by the test piece resting on a flat surface; third, the temperature can be easily controlled by partial immersion of the instrument in a water bath, all moving parts being made of bronze, stainless steel,[1] or monel metal, except the dial micrometer; fourth, the manipulation is simple, the movements of the indenting tool and weight being controlled by rotating a nut; and fifth, the use of a spherical indenting tool gives reproducible, sensitive, and accurate readings over a wide range of hardness. For quite soft materials, the sphere of $\frac{1}{4}$-in. diameter is replaced by one of $\frac{3}{4}$-in. diameter.

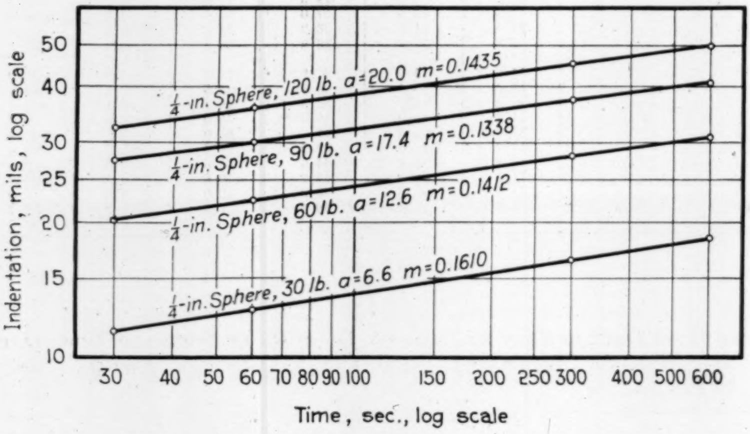

Fig. 2.—Indentation-Time Curves, 77 F. (25 C.).

Acknowledgment is made to Mr. E. E. W. Bowen, for the mechanical design of this instrument.

Many of the following data have been obtained by the use of a modification of the Riehle hardness tester for soft materials. The modification consisted in replacing the three lower $\frac{3}{4}$-in. balls by a flat bearing block and substituting a single ball of the desired diameter for the three upper balls. But on account of frictional losses and uncertainty of the correction for elastic deformation, the absolute indentations thus obtained are not particularly certain.

Effect of Time, Load, and Diameter of Sphere on Depth of Indentation

The observations recorded in the first four columns of Table I are typical of the behavior of asphalt tile under a constant dead load applied through a sphere, the tile resting on a flat surface. Other typical observations referring to spheres of various sizes and to various loads are given in Table II.

When the logarithms of the indentations of a given sample at a fixed temperature and under a given load applied by a given sphere are plotted as ordinates and the logarithms of the corresponding times as abscissas, the points lie on a straight line, as shown in Fig. 2. Hence the indentation, i, is related to the time, t, as follows:

$$i = at^m \qquad (1)$$

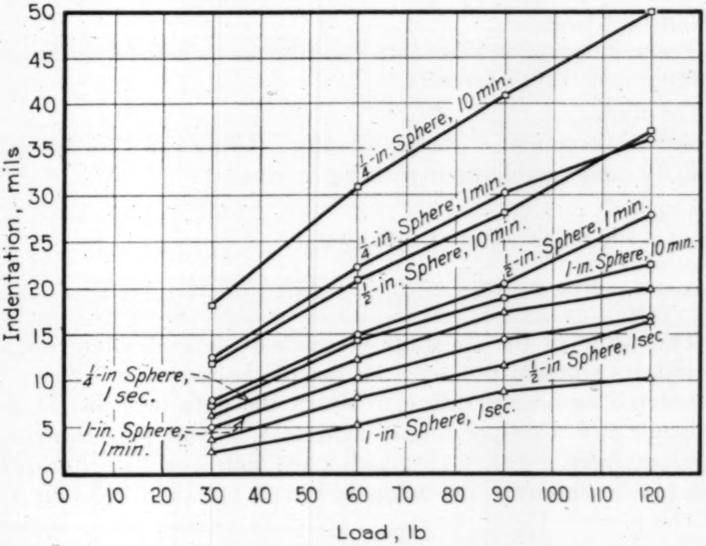

Fig. 3.—Indentation-Load Curves, 77 F. (25 C.).

The quantity a depends upon the load and the size of the sphere as well as upon the material and the temperature, but m probably depends only on the material and its temperature. The variations in its value in Table II are not large, and probably lie within the experimental error, as they are computed from the observed indentations at 60 and 600 sec. only.

If t is expressed in seconds and i in mils the indentation in 1 sec. under the condition of test is a mils. The value of i increases continuously with the time but at an ever decreasing rate, the rate of indentation at the time t being

$$\frac{di}{dt} = \frac{ma}{t^{1-m}} \qquad (2)$$

When the indentations at a given time are plotted as ordinates against the loads as abscissas, the observations given in Table II lead to Fig. 3. It is probable that elastic deformation is the explanation of the failure of these data to fall on straight lines passing through the origin. Hence, in the absence of more certain data to prove the contrary Devries' equation:[1]

$$i = Aw \tag{3}$$

where i is the indentation, w the load and A a proportionality constant, is taken to represent the relation between indentation and load at any constant time.

These indentation-load data can also be as satisfactorily related by Meyer and Hoyt's formula:[2,3]

$$i = Aw^n \tag{4}$$

in which the exponent n is close to unity. Using Eq. 3 for the sake of simplicity, and combining it with Eq. 1 gives:

$$i = bwt^m \tag{5}$$

Since a is the indentation at unit time

$$b = \frac{a}{w} \tag{6}$$

The value of the constant b seems to depend solely upon the material, its temperature, and the size of the indenting sphere. For the material and temperature to which the data in Table II apply, the values of b corresponding to spheres 1, $\frac{1}{2}$, and $\frac{1}{4}$ in. in diameter, are respectively 0.090, 0.133, and 0.193 mils per lb. For spheres $\frac{3}{4}$ and $\frac{3}{8}$ in. in diameter, the values of b are 0.116 and 0.163 mils per lb.

The general Eq. 5 certainly applies to asphalt tile. It has not been tried on other materials. Equation 1 which is used when the load and size of sphere is kept constant, holds for all materials so far examined. These include rubber, cork, linoleum, wood, brass, and a variety of bituminous materials.

APPLICATION OF EQ. 1 TO TESTING

Equation 1, when evaluated from two short-time measurements, 1 and 10-min. readings, for example, can be extrapolated to at least 24 hr. with experimental agreement as good at 24 hr. as is usually found between two 24-hr. tests. Table I serves as an illustration of the accuracy of a 24-hr. extrapolation from 1 and 10-min. readings.

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[1] R. P. Devries, "A Comparison of Five Methods of Hardness Measurement," *Proceedings*, Am. Soc. Testing Mats., Vol. XI, p. 709 (1911).

[2] Eugen Meyer, "Untersuchungen über Härteprüfung und Härte," *Mitteilungen über Forschungsarbeiten*, Verein Deutscher Ingenieure, Heft 65, pp. 1–61 (1909).

[3] S. L. Hoyt, "The Ball Indentation Hardness Test," *Transactions*, Am. Soc. Steel Treating, Vol. 6, No. 3, pp. 396–420 (1924).

These data were obtained by P. A. Sigler in the course of routine testing of Government samples. The 0.001 in. dial micrometer was estimated to 0.0001 in. It should be remarked that at one minute the hand of the dial is moving at the rate of approximately 0.0001 in. in 3 sec., and 0.0002 in. is about as close as a 0.001 in. dial micrometer can be read.

Since a in Eq. 1 is the indentation in mils at the end of 1 sec., a may be considered as a measure of the comfort of the floor. The constant m measures the tendency to indent with time. It should be emphasized that an indentation test consisting of a single measurement at the end of a short time, say 30 sec., gives very little information about the indentation behavior of an asphalt tile. Compare, for example, the indentation of two tiles, A and B, given in Table III. In service tile A failed by excessive indentation, whereas, tile B was satisfactory. The reason is that m is 0.25 for tile A and 0.14 for tile B.

TABLE III.—COMPARISON OF INDENTATION OF TWO SAMPLES.

	Indentation at 77 F. (25 C.), mils			Indentation at 115 F. (46 C.) at the end of 30 sec., mils
	30 sec.	1 min.	10 min.	
Tile A	6.0	7.1	12.7	24.0
Tile B	14.0	15.4	21.3	36.0

TABLE IV.—EFFECT OF VARYING EXPONENT m WHEN INDENTATION AT 30 SEC. IS 8 MILS. (INDENTATION IN MILS).

m	Log a	1 min.	10 min.	1 hr.	24 hr.	7 days	30 days	1 year
0.10	0.7554	8.6	10.8	12.9	17.7	21.6	24.9	32.0
0.11	0.7406	8.6	11.1	13.5	19.2	23.8	27.9	36.8
0.12	0.7259	8.7	11.4	14.2	20.8	26.3	31.3	42.2
0.13	0.7111	8.8	11.8	14.9	22.5	29.0	35.1	48.5
0.14	0.6963	8.8	12.2	15.6	24.4	32.0	39.3	55.7
0.15	0.6814	8.9	12.5	16.4	26.4	35.4	44.0	64.0
0.16	0.6668	8.9	12.9	17.2	28.6	39.1	49.3	73.6
0.17	0.6520	9.0	13.3	18.1	31.0	43.1	55.2	84.5
0.18	0.6372	9.1	13.7	19.0	33.6	47.6	61.9	97.1
0.19	0.6225	9.1	14.1	19.9	36.4	52.6	69.4	112.0
0.20	0.6077	9.2	14.6	20.8	39.4	58.1	77.7	128.0
0.21	0.5929	9.3	15.0	21.8	42.6	64.1	87.1	147.0
0.22	0.5781	9.3	15.5	22.9	46.1	70.8	97.5	169.0
0.23	0.5634	9.4	15.9	24.1	50.0	78.2	109.0	.....
0.24	0.5486	9.4	16.4	25.3	54.1	86.3	122.0	.....
0.25	0.5339	9.5	16.9	26.5	58.6	95.4	137.0	.....

Table IV was calculated to illustrate the effect of assuming different values of m for a tile of constant initial indentation (8 mils) at 30 sec. Table IV has been checked by laboratory tests up to the 24-hr. indentation. Agreements were of the accuracy shown in Table I. Experimental verification beyond 120 hr. is lacking. Some authentication of the long-time calculations is provided by the observation that floors in service are free from indentation if m is less than 0.15; are moderately indented, the degree depending upon severity of conditions of service, if m lies between 0.15 and 0.20; and are, as a rule, deeply indented if m exceeds 0.20. As a matter of interest, the effect of variation in m on the time necessary to indent completely through a $\frac{3}{16}$-in. tile was calculated for a load that produced an indentation of 8 mils in 30 sec., it being assumed that Eq. 1 is applicable to the entire indentation. It was calculated that 47,600,000 years would be required if $m = 0.10$, while only 105 days would be required if $m = 0.25$.

The December 7, 1933, revision of the proposed Federal Specification for Asphalt Tile controls m by using a table of 1 and 10-min. indentations, providing that if the indentation at 1 min. is a particular value, then the indentation at 10 min. shall not exceed the corresponding value given in the table. The relations are such that at one year calculated indentations shall not exceed 0.100 in.

Equation 2, which is the differential of Eq. 1, provides for the calculation of rates of indentation at any instant of time. The eighth and ninth columns of Table II give the time and indentations calculated by using the constants m and a shown in the fifth and sixth columns and setting di/dt of Eq. 2 equal to 0.001. Since i is expressed in mils, this is a rate of 0.000001 in. per sec. On the assumption that the rate of indentation is a function of the average pressure of the indenting tool, the tenth column was calculated by dividing the load (second column) by the spherical area of the corresponding indentation in the ninth column. The value in the tenth column shows a tendency to increase as the load is increased and as the size of the sphere is decreased. The irregularities in the variation with the load on a given sphere may arise from experimental errors, but the progressive change with the size of the sphere certainly does not.

If the same quantities (tenth column) are computed on the assumption that m is the same (0.145) in all cases, then they fall into three sets, one for each size of sphere, the members of any one set differing by not more than 5 units in the third significant figure; but the average of the three sets differs by more than two units in the second significant figure one from another.

From the geometry of a sphere it is evident that Eq. 1 cannot hold for indentations that are large with respect to the radius of the indenting sphere. Furthermore, when Eq. 1 is extrapolated from two short-time readings, the correspondence between observed and calculated readings at longer times though excellent when the observed indentations do not exceed one-third the radius of the indenting sphere, become unsatisfactory at greater indentations. At an indentation of one-half the radius, the observed indentations exceed the calculated indentations by 15 per cent. It has also been observed that when the indentations of two spheres of equal size, but unequally loaded, are compared on the same material, there is a linear relation between the two indentations for equal time intervals, until the indentation of the more heavily loaded sphere approaches one-half its radius, when its rate increases in comparison with the rate previously measured. The same thing occurs when equal loads act upon spheres of unequal diameters.

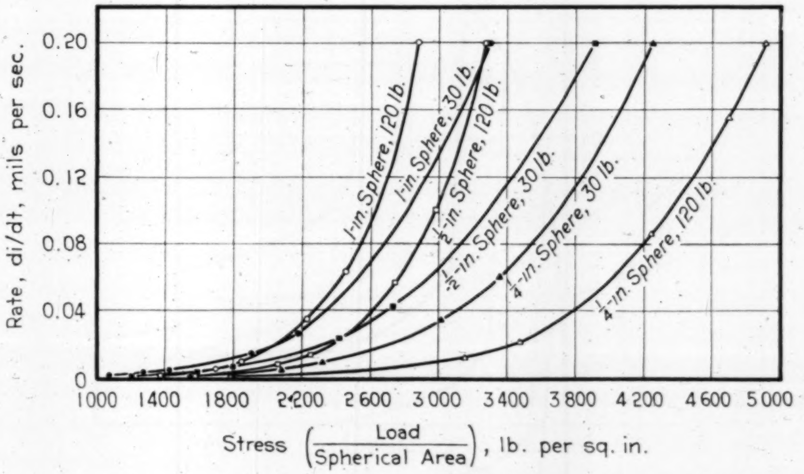

Fig. 4.—Relation of Rate of Indentation to Average Pressures, 77 F. (25 C.).

It would seem reasonable to assume that a geometrical shape could be selected which would start out like a sphere and have an "expanding radius" sufficient to correct the trouble that the finite radius of a sphere gives with Eq. 1. It is possible that a paraboloid of revolution is such a shape. Hence average pressures corresponding to those in the tenth column of Table II were computed on the assumption that the curved surface carrying the load is that of a paraboloid of revolution, the actual indentations being those in the ninth column. The values so computed are given in the last column. The paraboloid of revolution selected was that generated by rotating a parabola whose p, in the formula $y^2 = 2px$, equals the radius of the sphere actually used. For indentations small in comparison with the radius, such a paraboloid and sphere are identical within the accuracy of measurement. It will be observed that the values in the last column of Table II are much more nearly constant than those in the preceding column. A further correction could be made by correcting the 1 and 10-min. indentations for deviation of paraboloids from spheres. Inability to secure a paraboloid of revolution of the desired accuracy and small size prevented this theory from being tested experimentally.

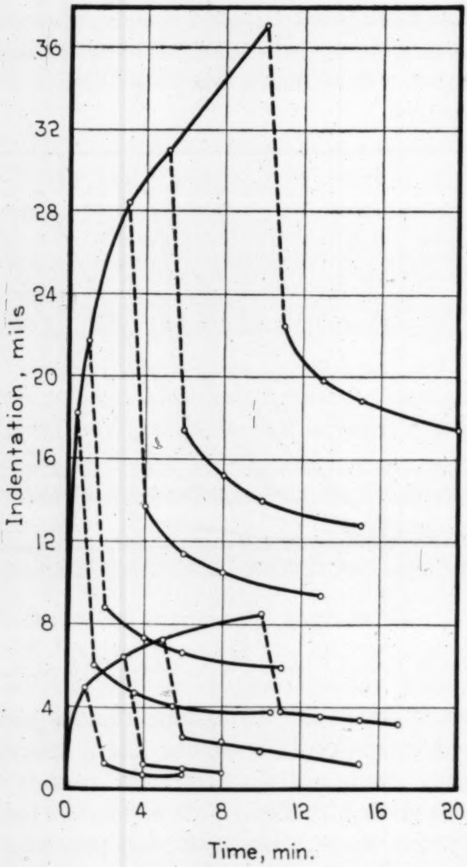

Fig. 5.—Indentation and Recovery of Two Samples of Asphalt Tile at 77 F. (25 C.). An 80-lb. load acted on a flat-ended pin 0.282 in. in diameter.

From the standpoint of a practical method of test, all that is necessary is that the load and size of sphere be selected so that the indentation will be small in comparison with the radius of the sphere.

A possibly useful application of the derivative of Eq. 1 consists in relating rates of indentation and average pressures as illustrated in Fig. 4 by some of the load and sphere combinations given in Table II.

The resemblance of these curves to the pressure-flow curves of Green[1] and others, will be noted.

These relations of depth of indentation, time, load, and diameter of indenting sphere with their derived average pressure and rates give promise of similar relations for other geometrical shapes of indenting tools. To date very fair agreement with spherical indentation has been found for horizontal cylinders (castors) based on the relation between average pressure and rate illustrated in Table II and Fig. 4. Flat-end pins have not acted as expected in that the rates of indentation are very much higher than for spheres with the same average pressure.

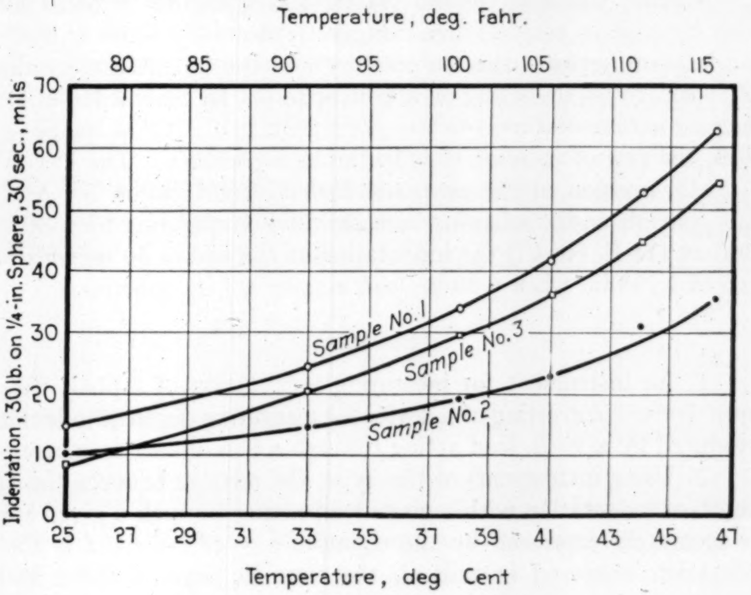

Fig. 6.—Indentation-Temperature Curves.

Recovery of Asphalt Tile

The preceding sections have dealt with indentation under a continuously applied load. Figure 5 gives some data on two samples of tile showing what happens when load is removed. The soft tile is a specimen much softer than would be commercially used. The hard sample complies with present specification requirements. The method of test consisted in permitting an 80-lb. load to act for varying periods of time upon a sample of asphalt tile, through a flat-end pin having a diameter of 0.282 in. The tile had previously been calipered at the point of contact of the pin with the tile. After removal of the loaded pin the thickness of the tile was again measured at time intervals as indicated. An immediate recovery of approximately a constant amount from the original indentation followed by further recovery at a decreasing rate is shown by these data.

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[1] Henry Green, "Further Development of the Plastometer and Its Practical Application to Research and Routine Problems," *Proceedings*, Am. Soc. Testing Mats., Vol. XX, Part II, p. 465 (1920).

EFFECT OF TEMPERATURE ON ASPHALT TILE

Asphalt tile is a thermo plastic and therefore becomes softer with increase in temperature. Figure 6 shows for some typical tile the relation between the temperature and the indentation produced at 30 sec. by 30 lb. acting on a $\frac{1}{4}$-in. sphere. In general the effect of increase in temperature is to increase indentation. As the temperature rises, the rate of increase of indentation accelerates. The December 7, 1933, revision of the proposed Federal Specification for Asphalt Tile controls undue softening with elevated temperature by specifying that at 115 F. (46 C.) the indentation at the end of 30 sec. shall not exceed 0.038 in. when a 30-lb. load acts on a $\frac{1}{4}$-in. sphere.

SUMMARY

1. An instrument for measuring indentation of asphalt tile has been devised, consisting of a device for measuring depth of indentation produced by a 30-lb. load acting through a $\frac{1}{4}$-in. diameter sphere.

2. Using instruments of this type, the relation between time and depth of indentation with a given load acting through a given sphere is accurately expressed by the equation $i = at^m$ where i is the indentation measured in mils, t, the time in seconds and a and m are constants.

3. In this equation the constant a is the indentation in mils at one second and may be taken as a measure of the comfort of the floor. The exponent m measures the rate at which indentation increases with time. For asphalt tile, values of m have been found as low as 0.10 and as high as 0.30. The upper limit for satisfactory service is 0.20.

4. The above equation also applies equally well to all other materials so far tested. These include cork, linoleum, rubber, brass, and wood.

5. When the indentation exceeds one-half the radius of the indenting sphere, the formula does not agree with the measurement. It is possible that a paraboloid of revolution would show better agreement for calculated and observed indentation over a wider range of indentation.

6. When the load is varied on the same size sphere, the relation between depth of indentation and load is expressed by the equation $i = Aw$ where i is depth of indentation, w is load and A is a constant.

7. When load is varied on the same size sphere, the relation between depth of indentation and time is expressed for asphalt tile by $i = bwt^m$ where i is depth of indentation, w the load on the sphere, t the time and b and m are constants.

8. In the above equation b appears to depend solely upon the material, its temperature, and the size of the indenting sphere, while m seems to depend solely upon the material and its temperature.

9. The immediate recovery of indentation produced by loads acting through flat-end pins is approximately a constant amount for a particular asphalt tile, followed by slow recovery at a decreasing rate.

10. Depth of indentation at constant time increases with increase in temperature. The rate of increase becomes greater for higher temperatures.

DISCUSSION

MR. W. H. HERSCHEL.[1]—We all have a tendency to give a property or characteristic some name, and then think we can define that characteristic by a single numerical value. This is not always possible. In the special case of a cube or a sphere we can define the shape and size by a single numerical value. In the special case of a simple viscous liquid, we can define viscosity by a single numerical value. But when it comes to a material, like these tiles, any combination of liquid and solid or any solid stressed above the elastic limit, we have a plastic material, and the consistency of a plastic material cannot be defined by a single numerical value.

I know of no case in the literature of a more beautiful example than is given in Mr. McBurney's paper of failure when the attempt is made to define consistency by a single numerical value and of success when two numerical values are used.

MR. J. W. WHITTEMORE[2] (*presented in written form*).—The results of Mr. McBurney's paper are especially interesting to me as they agree with some work that I have been doing on the plasticity of clays and mixtures of clays. About two years ago in trying to determine a satisfactory method for measuring the plasticity of clays, I applied a constantly increasing load through a $\frac{1}{2}$-in. flat-ended round shaft. With this arrangement, plotting the logarithm of the downward movement of the shaft as abscissas and the logarithms of the corresponding load as ordinates the points were found to lie on a straight line. In view of the difficulty of operation and at the author's suggestion I tried a constant load applied through a spherical surface. After using several different loads and spheres of different diameters, it was found that a load of 16 lb. and a sphere of 3-in. diameter gave the best results. It was also found necessary to lower the load onto the shaft as easily as possible to avoid any impact. This was done by floating the weight in mercury, draining the mercury off to lower the load onto the shaft.

The results of the tests on several clays indicated the same relation between load and indentation as described by the author. Plotting the logarithms of the depth of indentation as ordinates and the logarithms of the corresponding times as abscissas, the points were found to lie on a straight line with the relation $y = ax^m$ as described by the author.

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[1] Associate Physicist, National Bureau of Standards, Washington, D. C.

[2] Professor of Ceramic Engineering, Ceramic Engineering Dept., Virginia Polytechnic Institute, Blacksburg, Va.

It was found after testing several clays with various amounts of water, that the value of m remained fairly constant for the same clay although the value of a varied. This value of m ranged between 0.003 for a clay of low plasticity to 0.029 for a plastic ball clay. The value of a for any particular clay varied with the water content and undoubtedly is closely related to the water content of the clay.

If the plasticity of a material is considered as the force or pressure required to alter the shape of a plastic mass to a definite extent within a definite time, then, as the author intimates, this equipment and equation is applicable in measuring the plasticity of various materials.

MR. R. N. TRAXLER[1] (*presented in written form*).—The indentation test for asphalt tile which has been developed by Mr. McBurney is an empirical method which may be of value in testing mastics, pavements, etc., *in situ*. From research which we have been conducting on the rheological properties of asphalts and mixtures containing them, we have learned that the flow of bituminous materials is usually quite complex. Although we have not studied the operation of any instrument such as that described by Mr. McBurney we are reasonably certain that difficulties would be encountered in attempting to deduce the fundamental flow characteristics of the material from the data obtained with this apparatus. It appears that the indentation tester should be applicable for routine control tests on materials, the rheological properties of which do not vary appreciably from one batch to another. However, Mr. McBurney presents no data which show that the instrument is suitable for comparing fundamentally different materials.

MR. E. H. BERGER.[2]—Mr. McBurney by his definition, limits asphalt tile to compositions of asphaltic and/or resinous binders with asbestos fiber and raw cotton fiber. I see no reason for this. For a long time asphalt tile containing neither asbestos fiber nor raw cotton fiber has given satisfactory service. This definition also excludes waste cotton fiber, dyed cotton fiber, bleached cotton fiber, carpet flock, and materials of that kind, which cannot be considered raw cotton fibers but which give very desirable properties to asphalt tile. Roofing scrap also has been used for a long time and has given rather desirable properties to tile. Tile made with some of these excluded fibers have been used by Mr. McBurney for his test data, so why should they be excluded?

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[1] Research Division, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

[2] Consulting Chemist, Johns-Manville Corp., New York City.

Table I of the paper gives indentations ranging from 9.8 to 14.3. Is this an indication that the method and instrument are not applicable to indentations below 9.8 or above 14.3?

The instrument is a portable which is very advantageous. I think it should be brought out that the indentations depend to a large extent upon the sub-base on which the tile is laid. In describing the method some mention should be made of this and also of the most suitable support on which asphalt tile should rest during the test. I have known cases where the instrument has been used for testing tile supported on a base which indented easily and because of which higher indentations were observed on the tile which resulted in its being rejected. When the same tile was tested on a rigid non-indenting base much lower indentations were obtained which were within specifications. I believe this must be considered in using the instrument on tile installed on a job.

Mr. McBurney stated that the method and instrument are applicable to a wide variety of bituminous materials. I wonder whether he can give us some data on this, particularly with reference to the results on straight bituminous materials and those containing less filler than asphalt tile; for instance, straight asphalt, 70 parts of asphalt and 30 parts of filler by weight, equal parts of asphalt and filler, and 30 parts of asphalt and 70 parts of filler.

All the data given, with the exception of that in Fig. 6, are based on tests at 77 F. Would the same relations hold at 90 F., for instance, or for all temperatures up to the point where you get a very rapid break in the structure of the material? It might also be interesting to know whether m is the same at 115 F. as at 77 F.

MR. W. W. WERRING.[1]—We have done somewhat similar work on different materials, and I want to ask Mr. McBurney whether he has made tests on materials other than asphalt tile—for instance, rubber flooring or hard rubber. Hard rubber is interesting because it apparently has both plastic and elastic properties to a greater extent than asphalt tile. We would be very much interested in hearing of any information he might have on the comparison of asphalt tile, rubber tiling, and hard rubber.

MR. J. W. MCBURNEY.[2]—In answer to Mr. Werring's question, the method of test has been applied to both hard rubber and rubber floor tile. In comparison with asphalt tile, rubber is characterized by quite low values of m. For both hard and soft rubber, m is less than 0.01. The values of a range from 2.0 to over 40.0 mils depending upon the hardness of the rubber compound. An interesting application of the method is its use as a nondestructive test for distinguishing between hard rubber and asphaltic compounds in battery boxes.

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[1] Bell Telephone Laboratories, Inc., New York City.

[2] Research Associate of the Asphalt and Mastic Tile Assn. at the National Bureau of Standards Washington, D. C.

With reference to Mr. Berger's comments, I must disclaim responsibility for the definition of asphalt tile given in my paper. It is quoted from the current Federal specification for asphalt tile.

It should be emphasized that the use of the method and instrument is not confined either to asphalt tile or to asphalt tile of the range reported in Table I of the paper. This table was presented for the purpose of illustrating the degree of accuracy obtainable in routine testing.

Mr. Berger very properly emphasizes the necessity of supporting the test piece on an unyielding base. Large errors will be introduced otherwise.

As stated in the paper, both a and m are affected by temperature. A complete characterization of the plastic and elastic behavior of a material would involve the effect of temperature.

(*Author's closure by letter*).—For reasons which seemed good and sufficient at the time, the title, "Indentation of Asphalt Tile" was selected. With this title, it seemed proper to confine the presentation of data to the results of measurement on asphalt tile. For the information of those interested sufficient work has been done by the author and others to demonstrate that the equation $i = at^m$ holds for brass, maple wood, various pitches, several grades of coumarin resin, hard and soft rubber, linoleum, cork and a variety of asphalts and resinous compounds. There has been no systematic search for materials to which this equation does not apply. The random search conducted to date has not located such materials, although obviously the equation would not apply to any material such as portland cement mortar, which hardens with time.

It should be emphasized that from the author's standpoint the important feature is not the design of an instrument. It is the use of time as a variable. This permits the characterization of plastic behavior by two constants as pointed out by Mr. Herschel.

This is no disagreement with Mr. Traxler when he states that "the flow of bituminous material is usually quite complex." Using calculations such as are illustrated by Fig. 4 of the paper, the method affords a separation of bituminous materials into classifications of simple viscous liquids, non-Newtonian liquids and materials showing a yield value. This third type of materials would be called plastics, except for the definition of plasticity in the 1933 report of the Society's Committee E-1 on Methods of Testing.[1]

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 446 (1933).

A LABORATORY SERVICE TEST FOR PAVEMENT MATERIALS

By A. T. Goldbeck,[1] J. E. Gray,[1] and L. L. Ludlow, Jr.[1]

Synopsis

The need for a service test for pavement materials, which may be made conveniently and under controlled conditions of temperature, moisture, subgrade and traffic has become apparent. In the present paper, such a test is described. It consists of a 14-ft. diameter circular track in which pavement sections are laid and subjected either to the action of a roller or to a pneumatic tired wheel which may be equipped with tire chains.

Several tests are described to indicate the usefulness of the testing device. One of the tests was made to give an indication of the relative inherent stability of aggregates of various gradations. Still another investigation involved a determination of the value of stone screenings when used as a "blanket layer" under macadam to prevent upward intrusion of a clay subgrade into the stone. A third test had to do with a study of the effect of percentage of bitumen on stability of road mix surfaces. The stabilizing effect of a top dressing of chips when used with open type bituminous mixtures is discussed. Still another and quite different problem had to do with a study of the resistance of aggregates to crushing under the roller and under traffic. Finally, some preliminary tests of cold lay bituminous mixtures are discussed. These problems, which are quite diverse in nature, serve to illustrate the versatility of the testing device.

Introduction

The testing engineer in the highway field frequently is confronted with the problem of deciding on proper specification limits for various types of highway materials. At times, he feels the need of some form of rapid service test which would permit him to correlate service results with routine laboratory tests. When new bituminous mixtures are proposed for use in highway construction, he would like to have some means for determining rapidly whether these mixtures have sufficient merit to warrant their inclusion in specifications. The determination of the relative value of different materials for particular purposes sometimes becomes an important problem and a rapid and convenient means for solving it would be valuable. Many other problems arise in the laboratory which can be solved only by correlation of laboratory tests with service behavior.

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[1] Director, Bureau of Engineering; Laboratory Engineer; and Assistant Laboratory Engineer, respectively, National Crushed Stone Assn., Inc., Washington, D. C.

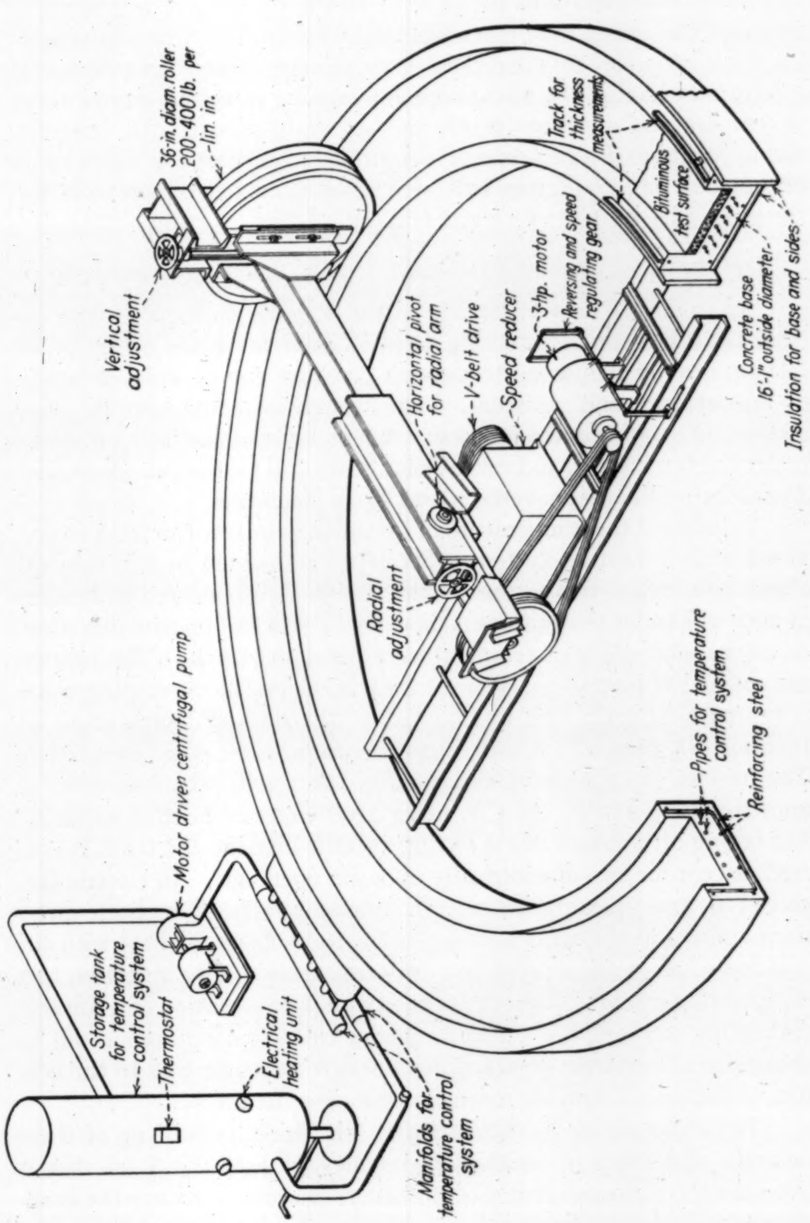

Storage tank for temperature control system
Thermostat
Electrical heating unit
Manifolds for temperature control system
Motor driven centrifugal pump
Vertical adjustment
36-in. diam. roller 200-400 lb. per lin. in.
Radial adjustment
Horizontal pivot for radial arm
V-belt drive
Speed reducer
3-hp. motor
Reversing and speed regulating gear
Track for thickness measurements
Bituminous test surface
Pipes for temperature control system
Reinforcing steel
Concrete base 16'-1" outside diameter
Insulation for base and sides

FIG. 1.—Isometric View of Circular Track Testing Apparatus.

These considerations led in 1931 to the building of a laboratory apparatus by which road materials might be subjected to actual service conditions, or to conditions very closely approaching those in service, but having the advantage not existing in actual service tests, of temperature, moisture, traffic and subgrade control. In designing the apparatus, the problem of determining the relative stability of bituminous pavement mixtures seemed of principal importance, but the possibility of other uses was realized.

DESCRIPTION OF THE CIRCULAR TRACK TESTING APPARATUS

An isometric view of the apparatus is shown in Fig. 1. The circular track is a concrete trough, 14 ft. in diameter at the center of the trough, heavily reinforced to prevent cracking due to wide variations in temperature and moisture. The trough is rectangular in cross-section, 18 in. wide and 6 in. deep. A 1-in. layer of insulating material under the track serves to prevent heat losses and to dampen vibrations. The sides are likewise covered with 1 in. of insulation.

To control the temperature of bituminous mixes, the track is provided with a heating system. Six pipes embedded in the concrete about $\frac{3}{4}$ in. below the bottom of the trough are connected to headers in such a manner that adjacent pipes carry flow in opposite directions to insure uniformity of temperature around the track. The headers are connected to a 30-gal. storage tank in which the circulating water is heated electrically and maintained at the desired temperature by thermostatic control. A centrifugal pump provides rapid circulation. The system is capable of maintaining the track, when covered, at approximately 140 F. (60 C.), using 5200 watts of heating capacity. For curing bituminous mixes having volatile binders, the track is covered, except for two diametrically opposite openings. An electric fan, placed in one opening, forces air around the track in both directions and out at the other opening. The combination of heat with circulation of air permits rapid and uniform curing. By connection to a refrigerating machine of proper capacity and the provision of a suitable circulating medium, low temperatures or freezing conditions could be obtained. At quarter points, pipes through the outer wall to the bottom of the trough provide means for draining the track.

The roller, 36 in. in diameter and 6 in. wide, is built up of three cast-iron disks each of which is 2 in. wide (see Fig. 2). Each disk is free to revolve independently of the others in order to reduce the dragging effect due to the travel in a comparatively small circle. The roller weighs approximately 200 lb. per in. of width. Driving is accomplished by the rotation of a radial arm about a vertical axis at the

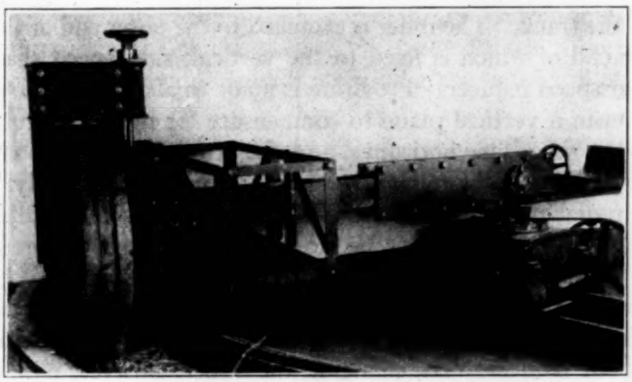


FIG. 2.—Apparatus with Roller Assembly.

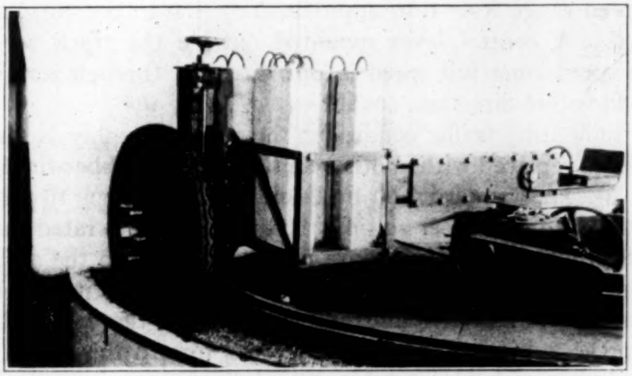


FIG. 3.—Apparatus Equipped with Rubber-Tired Wheel.

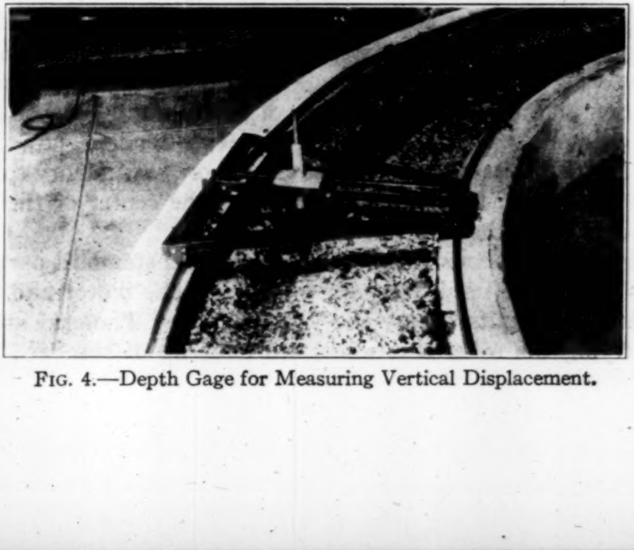


FIG. 4.—Depth Gage for Measuring Vertical Displacement.

center of the track. The roller is attached to the outer end of the arm, the inner end of which is fixed to the vertical, slow-speed shaft of a worm gear speed reducer. Provision is made to allow some movement of the arm in a vertical plane to compensate for minor irregularities. To keep the roller face horizontal and the arm level on various thicknesses of pavement, the roller assembly is adjustable vertically.

The radial arm is built of two telescoping sections and is adjustable in length to allow the roller to be moved in or out to cover practically the full 18 in. of width in the track. This operation may be performed while the machine is running. Power is applied by a 3-hp. reversible, variable-speed motor through V-belts and a countershaft to the speed reducer. By changing the V-belt sheaves, major steps in speed are obtainable; intermediate speeds may be obtained by varying the motor speed. The present combination of V-belts permits a speed range from 0 to approximately 450 ft. per min., peripheral speed. A control lever mounted outside the track serves to vary the speed from full speed in one direction through zero to full speed in the other direction.

For simulating traffic conditions the roller assembly is removed as a unit and replaced with another unit carrying a rubber-tired wheel (see Fig. 3). A standard 7.00 by 20-in. pneumatic bus tire is used, inflated to 50 to 55 lb. per sq. in. It is loaded to its rated capacity of 1900 lb. by placing weights in a basket attached to the arm. To prevent raveling action due to the drag of the tire as it travels in a small circle, it has been found necessary to tilt the wheel about 6 deg. from the vertical (the top of the wheel toward the center of the track) and to provide about 2 deg. "toe in."

In the design of this apparatus careful consideration was given to the advisability of using more than one wheel. It was decided, however, that the extra complication involved and the increase in power requirements and weight would not warrant this. The method of driving was also thoroughly studied. Because of the structural complication and attendant difficulties of control, and because of the fact that, despite tractive effort, road surfaces tend to shove in the direction of traffic, it was decided that the more satisfactory solution would be the simpler method of driving from the center of the track with the driving mechanism stationary.

Throughout the construction of the machine, standard parts and materials have been used wherever possible. The motor and speed reducer are stock units, as are the V-belt drives. The arm and the heads carrying the roller and wheel are built up of standard rolled shapes.

Auxiliary apparatus include a measuring device, or depth gage, for determining the thicknesses of the test sections, a spreader box, a screed, and a movable hoist for handling the roller and other heavy weights.

The depth gage is shown in Fig. 4. It consists essentially of a bridge spanning the trough and resting on strap iron rails mounted on the concrete curbing. Notched feet on the bridge fit over the rails and maintain its center line in a radial position. A slide moves radially across the bridge on guides in a horizontal plane and may be located by a scale attached to the bridge. A graduated vertical plunger is mounted on the slide. Thus, any point on the test surface may be located by the circumferential position of the bridge, the radial position of the slide, and the vertical distance as indicated by the plunger. The difference between the plunger reading previously taken on the concrete base and the reading on the test surface gives the actual thickness of the pavement. The rails on the curbing serve also to carry the spreader-box and the screed, thus facilitating accurate placing of material.

TABLE I.—GRADATION OF AGGREGATE USED IN ROLLING LOAD STABILITY TEST.

	$\frac{3}{4}$ to 2-in. Size	$\frac{3}{4}$ to $1\frac{1}{2}$-in. Size	$\frac{1}{2}$ to 1-in. Size	$\frac{1}{4}$ to 1-in. Size	$\frac{1}{2}$ to $1\frac{1}{2}$-in. Size	$\frac{1}{4}$ to $1\frac{1}{2}$-in. Size	$\frac{1}{4}$ to $\frac{3}{4}$-in. Size
Total retained on 2-in. screen,[a] per cent	2	..	..	..	..	..	..
Total retained on $1\frac{1}{2}$-in. screen,[a] per cent	38	2	..	..	2	2	..
Total retained on 1-in. screen,[a] per cent	74	63	2	2	47	38	..
Total retained on $\frac{3}{4}$-in. screen,[a] per cent	92	92	48	33	70	57	2
Total retained on $\frac{1}{2}$-in. screen,[a] per cent	96	96	93	63	93	75	47
Total retained on $\frac{1}{4}$-in. screen,[a] per cent	100	100	100	93	100	93	93

[a] Round openings.

USE OF LABORATORY CIRCULAR TRACK FOR THE INVESTIGATION OF ROAD MATERIALS

The circular track has proved to be a versatile testing machine for the study of road materials. Several investigations are described, primarily to show how this apparatus has been used to study different highway problems and to indicate its possibilities.

Tests of the Inherent Stability of Aggregates:

The purpose of this investigation was to determine the inherent stability of aggregates under the action of a roller. The variables were depth of surface constructed and gradation of the aggregate. Only stone aggregate is included in the present report.

It was believed that the action of the roller in causing rutting of the surface of a loosely laid layer of aggregate should be an index of the inherent stability of the material; thus, if a material of one gradation ruts deeper than another under similar action of the roller, it seems reasonable to infer that this material would be less stable than the other. The seven gradations shown in Table I were each tested as follows:

A uniform layer of stone was deposited on the concrete base over a length of about 11 ft., to form the test section. Initial thickness measurements were taken and the roller was run as follows: One passage over the inner 6-in. width, one passage over the outer 6-in. width, and one passage over the middle 6-in. width. The roller was then run over the middle 6-in. width for 5 additional passages and thickness measurements were taken after each passage.

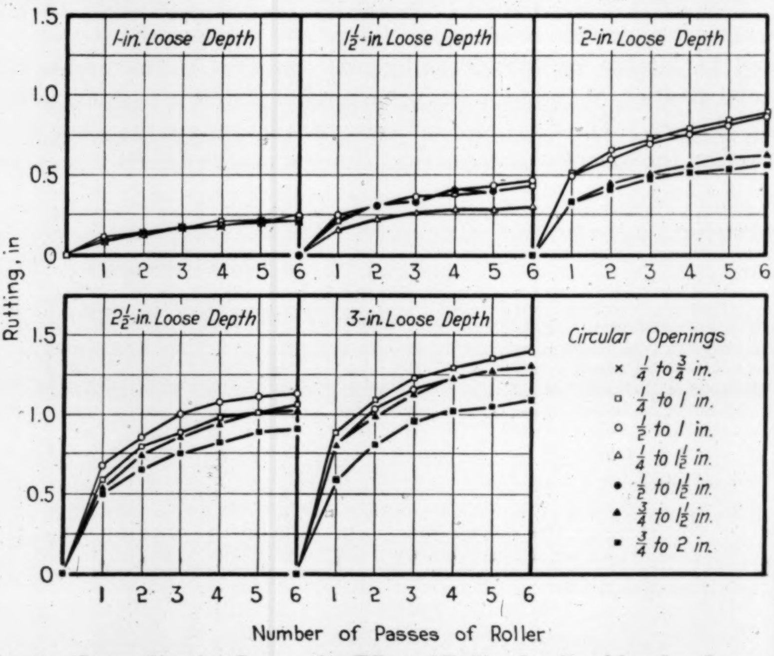


FIG. 5.—Curves Showing Compacting Effect of Rolling Load on Macadam Layers.

In Fig. 5 are shown the amounts of rutting of the various layers after the designated number of passages of the roller. These tests seem to indicate that the gradation of the aggregate influences the stability of a layer of material subjected to rolling loads and that stability of a thin layer may be increased by using a gradation in which the maximum size of the aggregate approaches the compacted thickness.

Stone Screenings as a Blanket Layer Under Macadam:

An experiment was performed to investigate the efficiency of stone screenings as a blanket course under a macadam layer. Two subgrades were prepared, each covering half the circumference of the track. One was made with fine plaster sand and the other with plastic clay. The clay was mixed with water to a consistency such that, after compaction by means of a tamp, it could be walked upon without greater indentation than that about $\frac{1}{4}$ in. The sand was moistened and tamped, but was of such a nature that it could not be compacted appreciably. The clay contained 17.1 per cent of moisture and the sand 7.5 per cent. On each of these two subgrades three sections were laid as outlined below:

Section	Subgrade	Blanket Course Stone Screenings	$1\frac{1}{2}$ to $2\frac{1}{2}$-in. Crushed Stone
No. 1	Clay	None	3 in. (loose)
No. 2	Clay	1 in. (loose)	3 in. (loose)
No. 3	Clay	2 in. (loose)	3 in. (loose)
No. 4	Plaster sand	None	3 in. (loose)
No. 5	Plaster sand	1 in. (loose)	3 in. (loose)
No. 6	Plaster sand	2 in. (loose)	3 in. (loose)

The following table shows the gradations of the screenings and of the $1\frac{1}{2}$ to $2\frac{1}{2}$-in. stone:

Gradation of Screenings		Gradation of $1\frac{1}{2}$ to $2\frac{1}{2}$-in. Stone	
Total retained on No. 4 sieve, per cent	0	Total retained on $2\frac{1}{2}$-in. screen, per cent	0
Total retained on No. 8 sieve, per cent	34	Total retained on 2-in. screen, per cent	59
Total retained on No. 16 sieve, per cent	60	Total retained on $1\frac{1}{2}$-in. screen, per cent	100
Total retained on No. 30 sieve, per cent	75		
Total retained on No. 50 sieve, per cent	84		
Total retained on No. 100 sieve, per cent	90		

The layers of screenings were placed loosely without any compaction and the stone layer was placed either immediately upon the subgrade or upon the loosely placed screenings to a loose thickness of 3 in. The roller was then run at a speed of approximately 3 miles per hr. in the same direction continuously around the track. The position of the roller was shifted laterally, about one inch for each round-trip. Approximately 60 passages of the roller were made over the track, during which time the roller was moved from the inside to the outside of the track and back again three times, making six

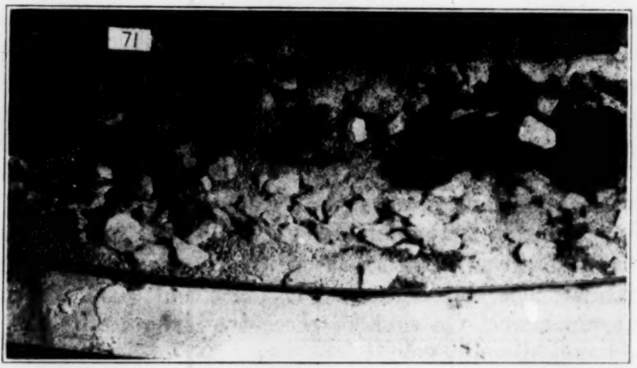


FIG. 6.—Sand Subgrade with 2 in. of Stone Screenings.

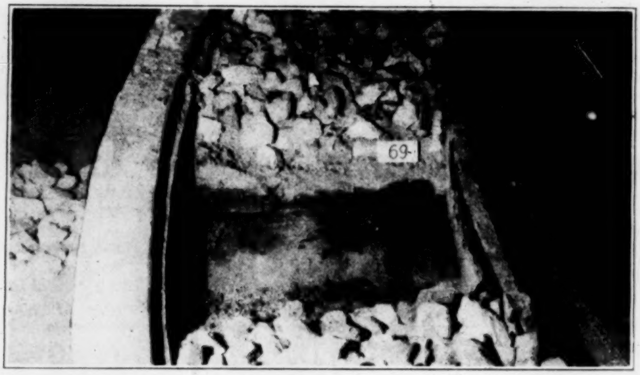


FIG. 7.—Clay Subgrade with 1 in. of Stone Screenings.

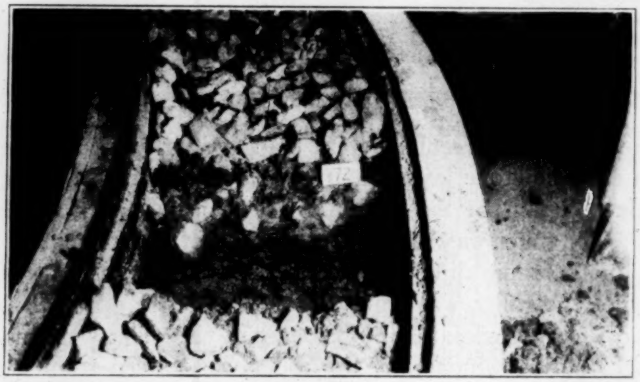


FIG. 8.—Clay Subgrade Without Blanket Layer of Stone Screenings.

complete lateral passages over each section. The effect of this action on the several sections is best seen in the accompanying Figs. 6 to 8.

In Fig. 6 is shown a surface view of the sand subgrade section containing 2 in. of screenings. The other two sand sections behaved in about the same manner as this one. The screenings layer was not effective in preventing the sand from working its way up into the stone.

In Fig. 7 is shown a cross-sectional view of the road surface having 1 in. of screenings on a clay subgrade. It will be noted that there is no upward penetration of the clay into the stone.

In Fig. 8 is shown a cross-sectional view of the section in which the stone is laid directly on the clay subgrade. Note how the stone has been pushed almost completely through the 3-in. layer of clay and how the clay has penetrated upward through the voids in the stone.

In conclusion, it would seem that when a macadam road is built on a clay subgrade, it would be a wise preacution to use a blanket course of screenings of 1 to 2 in. in thickness before laying the coarse stone. When the subgrade is composed of extremely finely divided clean sand, such as used in these tests, the screenings may not be efficacious.

Study of Effect of Percentage of Bitumen and Characteristics of Aggregates on Stability of Road-Mix Surfaces:

An investigation has been conducted in an attempt to show the effect of bitumen content on the stability of open type, road-mix pavements. A preliminary study of the suitable amount of bitumen to use for the crushed stone aggregate gave 3 per cent. The stone used had a bulk specific gravity of 2.72 and a percentage of wear of 3.3. The gradation for each of the sections was the same: namely, $1\frac{1}{2}$ to $\frac{1}{4}$ in., "straight line" gradation. The cut-back asphalt used was purchased to conform with the following specifications:

Flash point (Tag Open Cup)	80+ F. (27 C.)
Furol viscosity at 122 F. (50 C.)	200 to 400
Distillation, per cent by volume:	
Total distillate to 374 F. (190 C.)	....
Total distillate to 437 F. (225 C.)	10+
Total distillate to 600 F. (315 C.)	20+
Total distillate to 680 F. (360 C.)	35−
Tests on residue from distillation:	
Penetration at 77 F. (25 C.) 100 g., 5 sec	60 to 120
Ductility at 77 F. (25 C.)	60+
Soluble in carbon disulfide, per cent	99.5+

The road-mix pavement was constructed by following actual construction methods as nearly as possible. First, the stone was distributed by means of a spreader-box to a uniform, loose depth of about $2\frac{1}{2}$ in. Then, the track was divided into six equal sections, which were treated with a total of 2, 3, 4, $4\frac{1}{2}$, $3\frac{1}{2}$ and $2\frac{1}{2}$ per cent of cut-back asphalt in the order named. This order was used to eliminate any sudden change in the adjoining sections. The cut-back asphalt was sprayed in two applications of equal amounts. Immediately after spraying, the stone was thoroughly raked and turned over with shovels to simulate the action of a grader and to produce a uniform coating. The surface was then leveled with a strike-off board and allowed to cure.

After 24 hours of accelerated curing, followed by about 3 days' exposure to natural laboratory conditions, the surfaces were rolled. Immediately after rolling, the track was sprayed with $\frac{1}{10}$ gal. of cut-back asphalt per square yard, and stone chips, $\frac{1}{2}$ to $\frac{1}{4}$ in. in size and to the amount of 11.3 lb. per sq. yd. were broomed into the surface voids. The surface was rolled again; profile measurements were taken and the road was allowed to cure under natural laboratory conditions for 7 days.

TABLE II.—DEPTH OF RUTTING IN INCHES AFTER DIFFERENT NUMBER OF PASSES OF TIRE.

Cut-back Asphalt, per cent	Surface Dry			Surface Wet	
	2650 Passes of Tire	5300 Passes of Tire	7050 Passes of Tire	2750 Passes of Tire	5150 Passes of Tire
2	0.24	0.31	0.37	0.43	0.61
$2\frac{1}{2}$	0.20	0.29	0.33	0.41	0.65
3	0.19	0.26	0.31	0.43	0.55
$3\frac{1}{2}$	0.24	0.34	0.35	0.43	0.54
4	0.21	0.29	0.34	0.45	0.63
$4\frac{1}{2}$	0.24	0.34	0.38	0.49	0.63
Average	0.22	0.30	0.35	0.44	0.60

The test for the stability of the above described pavement was made by subjecting it to the action of the loaded tire and measuring the amount of rutting in the pavement sections after a definite number of passages. The pavement was maintained at an arbitrarily chosen constant temperature of 90 F. (32 C.) throughout the test.

After about 7000 passages of the tire an appreciable rut had developed. It was now decided to fill the voids in the pavement with water and to continue to run the tire. After 5150 additional passages of the tire, with the pavement wet, the rut had become so deep and rough that the test had to be stopped because of the vibrations set up in the floor of the building.

The wheel running over the pavement saturated with water has the effect of pumping the water through the voids, causing the asphalt to lose its adhesion to the aggregate. This last portion of the investigation may be considered as a durability test on this type of construction, especially when different aggregates and bituminous materials are thus compared.

It was apparent that the quantities of cut-back asphalt used in these test sections more than covered the range employed in this type of construction.

The results of these tests are tabulated in Table II.

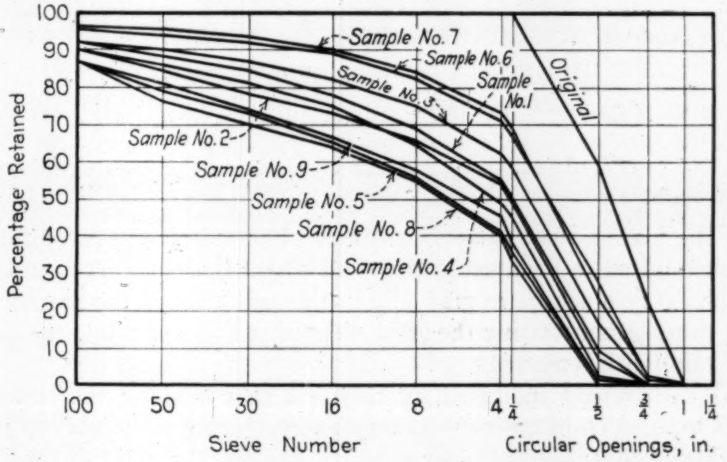


FIG. 9.—Gradation Curves Before and After Roller Tests on Aggregates for Bituminous Surface Treatment.

From these tests and for the particular type of construction studied, the indications are:

1. That bitumen content, within a practicable range has no appreciable effect on the stability of the open-type road-mix pavement.
2. That water greatly accelerates the disintegration of open-type, road-mix pavements under the action of traffic.

Stabilizing Effect of Top Dressing of Screenings on Open-Type Bituminous Mixtures:

In a particular study of the stability of open-type bituminous mixtures containing various percentages of crushed fragments, a rather important fact was brought out which further demonstrates the great usefulness of the circular track testing device.

A test pavement of $2\frac{1}{2}$ in. loose thickness was constructed in seven sections, each of the same gradation, $\frac{1}{2}$ to $1\frac{1}{4}$-in., round opening screens, and of the same asphalt content, namely, 3.3 per cent, but containing different percentages of crushed fragments. The track was rolled and cured for several days prior to testing under the action of the loaded tire.

After 120 revolutions of the wheel a rut developed of different depths for the seven sections but which averaged 0.19 in. in depth. In 189 additional revolutions, the rut averaged 0.33 in. in depth. All seven sections were comparatively unstable. The pavement was then re-shaped and 12.0 lb. per sq. yd. of screenings were rolled into the surface of each section. The screenings had the following gradation:

	TOTAL RETAINED, PER CENT
⅜ in. screen	0
No. 4 sieve	61
No. 8 sieve	78
No. 16 sieve	92
No. 30 sieve	94
No. 50 sieve	95
No. 100 sieve	97

The loaded wheel was then run for 4000 additional revolutions which resulted in an average depth of rut of 0.68 in. The rut developed very much more gradually in this test than in the preceding test, thus, demonstrating the great stabilizing effect of filling the surface voids with screenings.

It is strongly indicated by this test that choking the surface voids in an open bituminous mix with screenings greatly adds to the stability of that mix.

Resistance of Aggregates to Crushing Under the Roller:

An investigation has been made to determine the relative resistance to crushing of several aggregates for surface treatment work. Most of these aggregates passed the routine physical tests specified; yet it was known that some of them crushed excessively under rolling action during construction. The following samples were included in the test:

Sample	Material	Wear (Deval Abrasion Test), per cent	Toughness[a]	
			Core A	Core B
No. 1	Granite	2.6	8	16
No. 2	Granite	1.8	15	15
No. 3	"Greenstone"	1.7	12	15
No. 4	Limestone	3.5	5	5
No. 5	Granite	1.9	11	10
No. 6	Granite	1.7	24	25
No. 7	Limestone	2.9	7	7
No. 8	Granite	1.9	12	9
No. 9	Slag	Weight equals 78 lb. per cu. ft rodded		

[a] Cores A and B drilled at right angles to one another.

It was decided to apply the following simple roller crushing test:

Nine equal sections about 5 ft. long were marked off in the track. The identically graded samples were laid in the track in a 1-in. loose layer and the roller passed over them 200 times in a single path at approximately 175 ft. per min. To prevent excessive creeping of the samples, 100 passes of the roller were made, followed by 100 additional passes in the reversed direction. Thickness measurements were made before and after test, and mechanical analyses were made on samples taken from the path of the roller.

The gradations before and after test are shown in Fig. 9. It will be noted that there is a definite grouping of the samples. Samples Nos. 6 and 7 show high resistance to crushing; samples Nos. 1, 2, 3 and 4 are intermediate; and samples Nos. 5, 8, and 9 are inferior. This is also shown on Fig. 10 where the percentage reduction in thickness of the layer is plotted against the percentage of material passing the No. 4 sieve, the latter percentage being used as an index of crushing. Apparently there is no relation between the Deval abrasion test results and the resistance to the roller, nor does there appear to be any relation between the toughness test results and crushing under the roller.

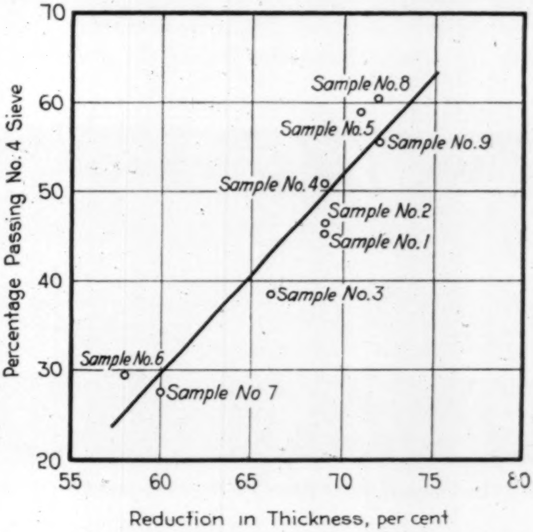


Fig. 10.—Percentage Reduction in Thickness of Layer of Aggregate Under Roller Action.

Figures 11, 12 and 13 show the appearance after rolling of a

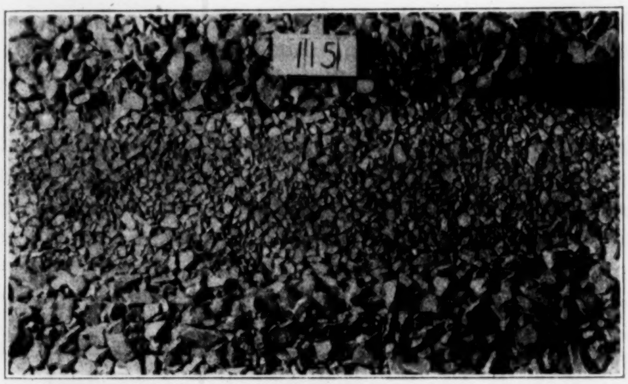


FIG. 11.—Crushed Stone After Rolling, Superior Quality.

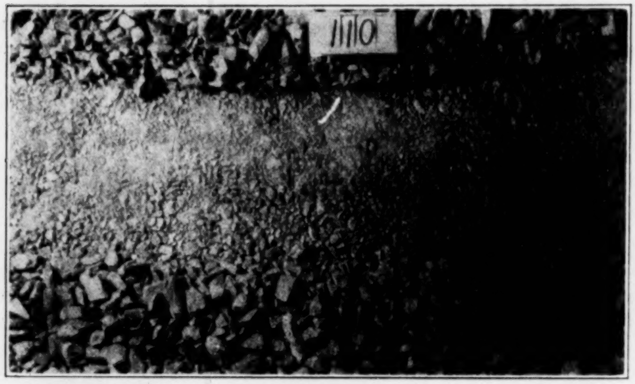


FIG. 12—Crushed Stone After Rolling, Intermediate Quality.

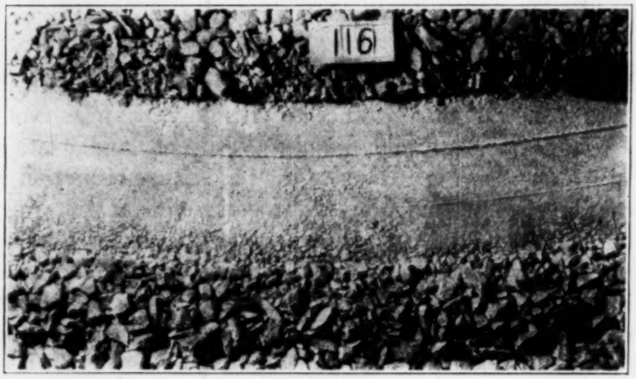


FIG. 13.—Crushed Stone After Rolling, Inferior Quality.

superior, intermediate and inferior material, respectively (Samples Nos. 7, 2 and 8).

The foregoing results show that the standard tests do not give an accurate indication of the resistance to crushing, and that a wide range of service results might be expected even with material satisfactorily passing the routine physical tests. It is believed that the roller test approaches actual service conditions and that the relative resistance to crushing so obtained is an indication of the relative merits of the materials for use where subjected to the crushing action of a roller during construction or of traffic.

Investigations on Cold Lay, Liquefier Type of Bituminous Mixtures:

Several problems have arisen in connection with the cold lay liquefier type of bituminous pavement. There seems to be no unanimity of opinion among engineers as to: (*a*) the gradation of stone for use in the base and top courses, (*b*) the most desirable type of liquefier, and (*c*) the most favorable proportions of the mix as between the several ingredients, including stone, asphaltic cement, liquefier and hydrated lime. The present investigations have been conducted primarily to obtain some indication of the best gradation of aggregate for the maximum stability under traffic and for the maximum durability under wet weather conditions involving rubber-tired traffic.

Some cold-mix plants ship the mixture into adjoining states and it is expensive and uneconomical to make widely varying mixtures in the same plant. Consequently, if there is a limited range of gradation which gives the most favorable result, it would be desirable to determine what that range is. Ease of handling and workability as well as serviceability are important considerations in mixes of this type.

Tests of Cold Lay Mixes.—The specifications of four adjoining states were analyzed and the limitations of gradation were determined for both the base and top courses. These ranges are shown in Table III, for the base courses and the top courses, respectively. Mixes were prepared in the laboratory by carefully grading the stone in accordance with these gradations, and mixtures were made up having the ingredients shown in Table IV.

For each of the four state specifications, three mixtures were made, involving the coarsest, the finest and the intermediate aggregate gradations. The intermediate gradation was made up as the average of the extremes of gradation. The coarsest top mix was used with the coarsest base mix in all cases and, similarly, the finest and intermediate bases and top courses were combined.

After laying and rolling the bases, they were cured until the free

TABLE III.—EXTREME GRADATIONS OF AGGREGATES POSSIBLE IN FOUR DIFFERENT STATE SPECIFICATIONS.

	Square Openings		Round Openings (¼-in. to 2-in. Screens)					
	State A		State B		State C		State D	
	Fine	Coarse	Fine	Coarse	Fine	Coarse	Fine	Coarse
BASE COURSE OF COLD LAY LIQUEFIER TYPE OF MIXTURES								
Total retained on 2-in. screen, per cent	..	..	0	0	0	0	0	0
Total retained on 1½-in. screen, per cent	..	..	13	32	5	25	18	32
Total retained on 1-in. screen, per cent	0	0	40	62	35	59	40	66
Total retained on ¾-in. screen, per cent	47	62	62	83	60	85	50	77
Total retained on ⅝-in. screen, per cent	73	95	..	..	..	..	..	..
Total retained on ½-in. screen, per cent	79	96	70	87	69	90	65	92
Total retained on ¼-in. screen, per cent	93	96	83	93	85	99	73	92
Total retained on No. 10 sieve, per cent	95	97	87	95	89	99	80	94
Total retained on No. 30 sieve, per cent	97	98	93	96	94	99	85	96
Total retained on No. 80 sieve, per cent	99	99	98	99	98	100	89	97
TOP COURSE OF COLD LAY LIQUEFIER TYPE OF MIXTURES								
Total retained on 1-in. screen, per cent	..	..	..	..	..	..	..	0
Total retained on ¾-in. screen, per cent	..	..	..	0	..	..	..	3
Total retained on ⅝-in. screen, per cent	0	0	0	4	0	0	..	5
Total retained on ½-in. screen, per cent	14	22	12	20	10	16	0	7
Total retained on ¼-in. screen, per cent	50	80	63	85	55	85	37	65
Total retained on ⅛-in. screen, per cent	91	95	..	..	..	..	..	..
Total retained on No. 10 sieve, per cent	92	96	74	89	67	89	70	86
Total retained on No. 30 sieve, per cent	96	98	85	94	82	94	80	92
Total retained on No. 80 sieve, per cent	98	99	97	99	97	99	88	95

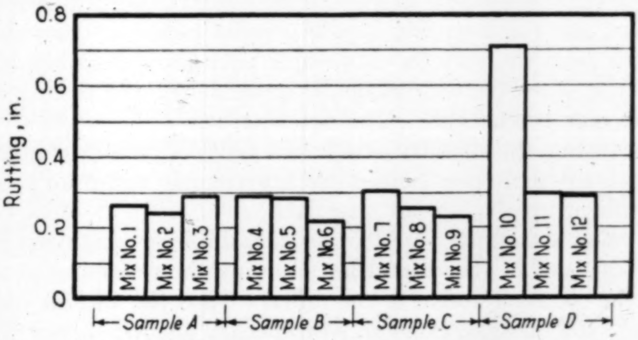
TABLE IV.—REQUIREMENTS FOR COLD LAY LIQUEFIER TYPE BITUMINOUS CONCRETE IN FOUR ADJOINING STATES.

Fine, Medium and Coarse Gradations.

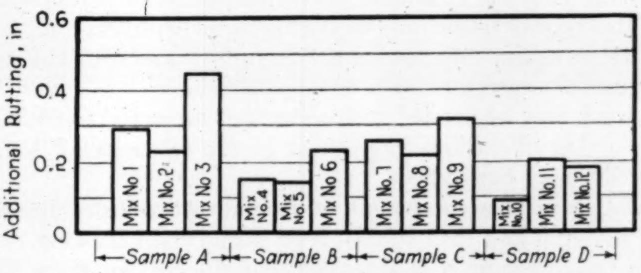
	State A			State B			State C			State D		
	Section No. 1	Section No. 2	Section No. 3	Section No. 4	Section No. 5	Section No. 6	Section No. 7	Section No. 8	Section No. 9	Section No. 10	Section No. 11	Section No. 12
BASE COURSE												
Aggregate, per cent	92.0	93.55	95.1	92.2	93.75	95.35	93.5	95.0	96.6	91.2	92.9	94.6
Liquefier, per cent	1.0	0.7	0.4	1.0	0.7	0.35	1.5	0.95	0.4	1.3	1.1	0.9
Lime, per cent	1.0	0.75	0.5	1.0	0.75	0.5	1.0	0.75	0.5	2.5	2.0	1.5
Asphalt cement, per cent	6.0	5.0	4.0	5.8	4.8	3.8	4.0	3.3	2.5	5.0	4.0	3.0
TOP COURSE												
Aggregate, per cent	90.0	92.05	94.1	91.0	92.57	94.15	91.5	93.3	95.1	89.2	90.5	92.1
Liquefier, per cent	1.0	0.7	0.4	1.0	0.68	0.35	1.5	0.95	0.4	1.3	1.1	0.9
Lime, per cent	1.0	0.75	0.5	1.0	0.75	0.5	1.0	0.75	0.5	2.5	2.0	1.5
Asphalt cement, per cent	8.0	6.5	5.0	7.0	6.0	5.0	6.0	5.0	4.0	7.0	6.25	5.5

liquefier was driven off. The top courses were rolled and cured in a similar manner. The test pavement was then subjected to the loaded tire running in a single path.

Results.—The results, expressed in terms of the average amount of rutting which occurred in the various sections, are shown in Fig. 14. It is noticeable that a wide range in stability existed. Mix No. 10 was particularly unfavorable. It was finely graded and was the heaviest and densest mix in the various sections. There is not much to choose in stability in the remaining sections as judged by the amount of rutting produced under dry conditions. Sections Nos. 3, 6, 9 and 12 contain the most open mixes in the respective specifications and under wet conditions sections Nos. 3, 6 and 9 seem to be the least durable and section No. 10 the most durable. Sections Nos. 4, 5 and



Rutting Due to 5000 Passes of Wheel Equipped with 7.00 x 20 Tire Under 1900-lb. Load with Pavement Dry



Additional Rutting Due to Additional 10,500 Passes of Wheel with Pavement Wet

FIG. 14.—Curve Showing Relative Stability and Durability of Cold-Lay Liquefier Type Mixes.

NOTE.—Additional rutting measurements were taken at the outside portion of the rut where ravelling was most pronounced.

6 were the most resistant and best sections considering both stability and durability.

The above tests are preliminary and are only a small portion of a more comprehensive series of tests on cold lay mixtures. These tests are too extensive to be considered further at the present time.

CONCLUSION

It was not the intention in the present paper to draw definite conclusions from the various tests which have been described briefly, but rather to show the wide range in applicability of a testing device of the circular track type. The machine was built originally as a device for testing the stability of different types of pavement mixtures, but it has many more possibilities and is extremely useful for studying other problems involving road materials.

The roller attachment approaches actual rolling conditions during construction, and is very useful for studying the resistance of aggregates to the crushing action of the roller. Thus the various physical tests conducted on aggregates may be checked against service results. Since the action of the rubber-tired wheel is similar to that of traffic, service tests for stability can be made. Furthermore, by the use of chains on the tire, the resistance of various kinds of surfaces to the action of this type of traffic may be studied so as to obtain comparative results. Here, again, such results are useful in correlating the regular physical tests of aggregates with service behavior.

The track may be flooded with water and subjected to traffic and, thus, the action of various aggregates in the retention of their bituminous film may be studied.

Finally, with this equipment, it is quite possible to freeze the surfacing material and then subject it to traffic upon thawing.

From the foregoing it is apparent that the circular track testing machine is an excellent tool for quickly coordinating laboratory physical tests with service behavior and for determining rapidly the service value of highway materials and their combinations.

DISCUSSION

Mr. M. H. Ulman[1] (*presented in written form*).—We were so favorably impressed with the possibilities of the circular track machines developed by the Bureau of Public Roads and Mr. Goldbeck as adaptable equipment for accelerated laboratory tests in the studies of subgrade stabilization, behavior of aggregates and stability of bituminous mixtures under service, that we have constructed a somewhat similar one. The principal change in design compared to Mr. Goldbeck's machine was to incorporate a direct drive of the wheel for more nearly simulating motorized vehicular behavior.

I can appreciate in a study of the authors' paper that they were desirous of giving information relative to the possibilities of the machine as a tool for the testing engineer rather than to furnish a detailed discussion of each of the investigations. I wish, however, to submit the following comments on each of the topics covered:

Tests of the Inherent Stability of Aggregates.—No information is furnished in the paper as to the kind of stone aggregate used. Presumably it covers only the effect of gradation on one type of aggregate. In accordance with our observations sandstones, trap rock and limestones behave differently under the roller, the shape and quality of the fragments having an important bearing on the results obtained. This machine should furnish valuable data on these variables.

We have verified the authors' conclusions under actual service, for we always use an aggregate gradation in the various types of flexible bases and surface construction in which the maximum size very closely approaches the compacted thickness for the most durable results.

Study of Effect of Percentage of Bitumen and Characteristics of Aggregates on Stability of Road-Mix Surfaces.—It is not stated whether the percentages of bitumen are based on volume or weight, but I assume the latter to be the case, and also whether the percentage is on the basis of the cut-back or actual asphalt cement content of the cut-back.

The stability of this type mixture would also be partially affected by the shape of the crushed fragments. We have made studies of the effect of water on these type surface courses and find that the influence may vary with the type of bitumen, for certain bitumens may be more resistant to the water action than others. Such investigations should accordingly not only cover the bitumen content but the shape of particles and type of bitumen. The influence of cold weather by freezing and thawing is another factor which is important, for certain bitumens are more susceptible and thereby become brittle at the colder temperatures.

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[1] Assistant Engineer of Materials, Pennsylvania State Highway Dept., Harrisburg, Pa.

The type of base and shoulders used also has an important influence on these fine-graded open mixes, as rigid bases will hold the water on their surfaces while the flexible type bases permit of better drainage.

Stabilizing Effect of Top Dressing of Screenings on Open-Type Bituminous Mixtures.—A mixture of the type described would have little durability unless a seal coat was used in the construction consisting of choke stone and bitumen, in order to waterproof the mixture. We favor the use of stone chips free from dust rather than screenings as described by the authors of the paper, for the dust of fracture may have a tendency to "ball" when the bitumen is applied and also to prevent proper adherance of the bitumen to the stone. We use a double instead of a single seal coat application for the most desirable results. The results obtained by the authors of this paper substantiate the knowledge gained by field studies and again proves the value of the machine.

Resistance of Aggregates to Crushing Under the Roller.—The machine gives a practical means for evaluating the behavior of stone under the roller. We have sources of stone supply in Pennsylvania from which we cannot approve the aggregates for macadam or surface treatment chips due to excessive crushing taking place during construction. These particular sources, however, meet our requirements for physical tests, for the percentage of wear is less than 5 and toughness in excess of 6. This confirms the authors' conclusion that "the standard tests do not give an accurate indication of the resistance to crushing."

The machine also will give information as to the behavior under the roller of crushed fragments, which may be detrimentally affected by the shape of the particle, cleavage planes and laminations. The results, which would be seriously affected by these conditions, cannot be ascertained by the generally used tests, but a roller test gives correct information as to the aggregate's behavior under service.

Investigations on Cold Lay, Liquefier Type of Bituminous Mixtures.—The important considerations for these type mixes are ease of handling and workability together with durability. It is unfortunate that some promoters have stressed the ease of handling and workability in the composition of their mixtures at the probable expense of durability. The ease of handling and workability is a function of the amount and type of liquefier. Excess liquefier and one which lacks volatility will cause subsequent bleeding and rutting under traffic regardless of aggregate gradation. The authors' results again confirm that the coarsely-graded open mixes give the greatest durability, other conditions being favorable, such as proper quantity and type of liquefier. This type of surface course as originally designed provided for an open bottom course having large void spaces to provide for the top course, which filled the voids and sealed the underlying bottom course and thereby obtaining a monolithic two-course pavement.

We have pavements of this type under service since 1907 on highways carrying heavy traffic and such pavements are in excellent condition. The circular track is ideal equipment for making laboratory investigations as to the effect on durability of the variables already referred to and also relative to the amount of additional compression this type of mixture will take under traffic and the varying temperature conditions. It is generally known that these pavements will compress more in warm weather than in cold and will continue to compress under the action of traffic.

This equipment should be an invaluable aid to the testing engineer in view of its many applications. It probably will serve as a measure for evaluating the merits of the various types of bituminous mixtures now being promoted.

MR. PRÉVOST HUBBARD.[1]—One thought that occurred to me had to do with results reported in the paper on open type aggregate mixtures with a wide variation in percentage of bitumen. These variations of from 2 to $4\frac{1}{2}$ per cent of bitumen in the mixtures actually represent a variation in the bitumen content itself of about 125 per cent. The conclusions, as far as they went, were that the percentage of bitumen within these limits had no noticeable effect upon stability. My comments in this connection are not a criticism of the conclusions drawn, because undoubtedly what the authors found was true for the temperature range of the tests. It would be unfortunate, however, for the reader to come to any conclusion that this variation in percentage of bitumen is not a very important matter from other standpoints than that of stability at or around 90 F.

I believe there has been a regrettable tendency in recent years to cut down the percentage of bitumen in these open type mixtures and also in some of the low cost closed type mixtures to a minimum which is exceedingly dangerous and which has resulted in more serious deterioration of the highway, particularly under low temperature winter conditions, than would have been the case if the film thickness or percentage of bitumen had been increased. The average reader might come to the conclusion that 2 per cent bitumen would be all that would be necessary for this open type mixture, and this I believe is absolutely erroneous. A mixture containing such a low percentage of bitumen with such a thin film thickness would disintegrate much more rapidly under severe winter conditions than one containing the maximum percentage.

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[1] Chemical Engineer, The Asphalt Institute, New York City.

MR. ULMAN.—The bituminous surface course to which I referred as having been placed in 1907 and which is still in good condition contained approximately 4.5 per cent asphalt cement in the bottom course mixture and approximately 6 per cent in the top course mixture. This will confirm Mr. Hubbard's statement relative to the necessity for obtaining sufficient bitumen content.

MR. J. S. MILLER, JR.[1]—I should like to say just a word that may indicate another line of thought in the usefulness of this track and at the same time clarify the statement that was made in other comments upon this paper, namely, the question of the effect of water on a mixture under test. Certain work we have been doing in our laboratories and which has been referred to by others, indicates that a very definite factor in this water effect upon mixtures is the selective wetting of aggregates as between water and bitumen. Perhaps this or some other type of track may be a tool by which we can study interfacial tension between aggregates and bituminous substances with respect to any difference in wetting power for that aggregate between bitumen and water.

MR. F. H. JACKSON.[2]—The work at the Bureau of Public Roads so far has been confined to determining the relative stability of various bituminous mixtures when subjected to the laboratory service test to which the authors refer. We have not as yet gone into the question of determining the relative amount of crushing which takes place when various types of aggregates are subjected to the test.

There is one point I should like to mention in connection with the work that the authors have done, which has not been touched upon. The test which they developed is of course not adapted to routine laboratory testing. The equipment is too complicated and costly for that. It is a research tool, an accelerated service test which measures the relative resistance of various aggregates to crushing and in this way furnishes information as to their relative value for use as surface treatment materials. What is needed now, it seems to me, is the speedy development of a routine laboratory test, the results of which can be correlated with an accelerated service test of this type. If such a test can be developed, it may be used to replace the Deval abrasion test, which seems to be pretty largely discredited as a result of this work. Recent work with the Los Angeles rattler test used by the California Highway Department indicates that this method may prove satisfactory.

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[1] Director, Technical Bureau, The Barber Asphalt Co., Maurer, N. J.

[2] Senior Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

MR. W. S. HOUSEL.[1]—There are several points in this paper that are very interesting to the Michigan State Highway Department as we have a very difficult problem in finding crushed stone within the State that meets the specifications for bituminous aggregates. The authors apparently consider 7 as being a low toughness value. We feel rather pleased when we find a stone with a toughness as high as 7 and at the present time there is only one producer within the State who can furnish material which is consistently above this lower specification limit. Under these conditions we are, of course, interested in the development of a more suitable laboratory test for such materials and would be willing to accept changes in the type of tests used if it can be shown that the tests now in use are not reliable criteria.

At the present time we are conducting an investigation on the behavior of various aggregates used in pavement construction along lines which almost exactly parallel the tests outlined by the authors. The investigation is being conducted by Mr. Rothgery of the Michigan State College at Lansing. The Michigan State College Laboratory has installed a circular roller of practically the same design as that described. The accompanying Fig. 1 is a photograph of the apparatus. The track is 16 ft. in outside diameter with a testing trough of rectangular cross-section of variable depth and 2 ft. in width. The roller is equipped with 3-speed transmission with automatic control which allows operation forward and reverse at variable speeds. The roller is also equipped with a worm gear which makes it possible for the rotating roller to travel the full width back and forth across the trough where the sample is placed. We feel that this arrangement is desirable to more closely approximate actual traffic conditions.

The investigation now under way provides for the roller test on not only crushed stone but slag and gravel as well, these tests being made on the loose material to give a comparative measure of the resistance to crushing and abrasion of these typical aggregates. Samples of the same material are being subjected to several commonly used abrasion tests, such as the standard abrasion, modified abrasion, and the Los Angeles rattler. We feel that the roller test is perhaps more closely akin to the actual service condition of the material and it is our hope to correlate our results with the tests now in common use. We are also planning to make tests on the same aggregates when combined in the representative bituminous mixtures, again measuring the resistance of the aggregate itself and the stability of the final road surface.

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[1] Research Consultant, Research and Testing Division, Michigan State Highway Dept., University of Michigan, Ann Arbor, Mich.

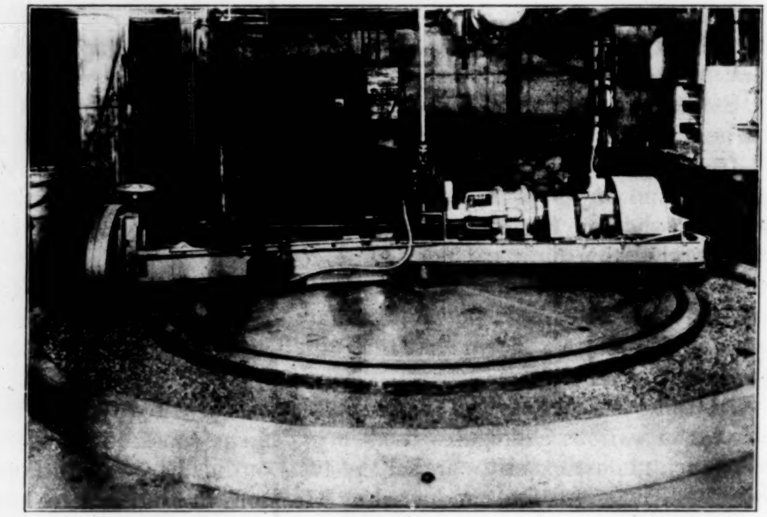

FIG. 1.—Apparatus Used by Michigan State Highway Department.

We are in agreement with Mr. Jackson's statements that this type of test is a research tool and is not particularly feasible for a routine laboratory test. The machine is rather expensive and occupies a very considerable amount of space. While this test may reproduce much more accurately actual service conditions it seems that the correlation of test results with present standard tests may be the most desirable improvement which might result from the investigations that are being made.

In the tests conducted so far we have had considerable difficulty, particularly with gravel, due to shoving and displacing of the material from beneath the roller. When the width of the roller is considerably less than the width of the track the material ruts so quickly that we feel the test is not a true indication of the behavior of a particular thickness of material being tested. We are now planning on making the roller the full width of the trough to eliminate the lateral displacement. I should like to ask Mr. Goldbeck what he thinks of the idea of having the material entirely confined and whether or not this condition would more closely approximate the condition which the material encounters in service.

MR. A. T. GOLDBECK.[1]—I rather like it. I think that is an improvement over the narrow roller. We find when we use a 6-in. width of roller on a round type of aggregate, that we do get a lateral shoving as well as a longitudinal shoving. I think, however, that you will find the rounded material will shove more longitudinally than angular aggregates even with the full width of roller. A roller of 3-ft. diameter produces a higher intensity of pressure on the road surface than would a roller of greater diameter, and for that reason we find more difficulty in rolling a bituminous surface than we would find were the roller of greater diameter. I think if I were going to re-design the machine I might do two things—increase the diameter and width of the roller.

MR. W. H. FULWEILER.[2]—I should like to ask whether it is Mr. Goldbeck's opinion that the tests made with the dry cover stone would give results in the same direction as would be secured in an actual bituminous surface where the particles are supported by the bituminous material.

We have noticed in going over our own results that when the steel tired traffic practically disappeared from our roads as it did five or six years ago there was quite a change in the grading curves in the aggregates of the surface mixtures, and that the present grading is quite different from that of ten or fifteen years ago. Some of this change has undoubtedly been due to changes in the grading of the original cover stone, but we believe that much of it is due to the change in the type of traffic that our surface materials are called upon to withstand. I will be glad to know whether Mr. Goldbeck has any figures to show the difference in the grading of materials tested dry, and in bituminous mixtures under the same loading conditions.

MR. GOLDBECK.—I think Mr. Fulweiler is perfectly correct. You can get a high stability, maybe even a higher stability with aggregates graded to a fine size with a certain percentage of bitumen than with coarse aggregates with still another percentage of bitumen. Of course, film thickness plays a big part in the stability of bituminous mixtures.

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[1] Director, Bureau of Engineering, National Crushed Stone Assn., Inc., Washington, D. C.

[2] Chemical Engineer, United Gas Improvement Co., Philadelphia, Pa.

These original tests on stone alone without any bituminous material added were made without any particular reference to what would be the effect had we added bituminous material. This was simply an investigation that we made to determine something of the inherent stability of different layers of different kinds of materials when not mixed with any kind of bituminous material. The results have a bearing on the question of what happens to railroad ballasts and, perhaps, what happens to loose layers of stone under traffic. They have something to do with the difficulty of rolling aggregates having different shapes and different sizes.

THE TESTING OF RUBBER AND RUBBER-LIKE MATERIALS FOR OIL RESISTANCE

By O. M. Hayden[1]

Synopsis

The serviceable life of rubber and rubber-like compositions in contact with oils and solvents can be accurately forecast only if laboratory tests are conducted under conditions comparable to those that will be encountered in service.

It is shown that changes in weight and volume during immersion in oils are less important than changes in such physical properties as resilience, strength, toughness, etc.

At present the oil resistance of rubber and rubber-like materials cannot be adequately evaluated from tests that are expressed numerically. The most significant changes that occur during immersion in oils take place in such physical properties of the compound as flexibility, toughness, surface disintegration, and the like. As yet these changes are evaluated by superficial examination rather than by mechanical tests. It is most unfortunate that this is true, because these are the really important properties that determine the suitability of any given composition for use under any specified conditions of service.

Methods of determining the tensile strength, extensibility, elasticity, abrasion resistance, cutting resistance, and other physical properties of vulcanized rubber are well established. However, less attention has been given to the development of methods of determining its oil resistance and interpreting the results of such tests.

As applied to rubber and rubber-like products the term "oil resistance" is defined to mean their capacity to resist physical changes during or following contact with oils or solvents. The purpose of this paper is to discuss methods for determining the extent of the various changes that occur and to comment on the significance of these changes as related to the performance of the product in service.

Significance of Dimensional Changes:

The most obvious effect of immersing rubber or rubber-like materials in oils or solvents is the increase in volume. This is, of course, an important property, but it is of less importance than would be inferred by reading many consumer specifications. Rubber is used as a material of construction because of its extensibility, elasticity, strength, and toughness. If none of these properties were affected by contact with oil, changes in dimensions and in volume would not greatly restrict its use. Obviously, the tendency to increase in volume is a serious shortcoming in rubber that is used for the tube of small diameter oil or solvent hose, but if this were the only shortcoming it could be taken care of by building the hose with a sufficiently great internal diameter to permit of sufficient flow after the rubber had reached its maximum degree of swelling. The percentage of increase in thickness and in volume is of little value in indicating the life of a rubber product, as will be seen from an analysis of the test results shown in Table I on two rubber compounds.

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[1] Manager of Rubber Laboratory, Rubber Chemicals Division, Organic Chemicals Dept., E. I. du Pont de Nemours and Co., Inc., Wilmington, Del.

TABLE I.—EFFECT OF KEROSINE ON TWO RUBBER COMPOUNDS.

	Cure	Percentage Increase in Weight	Percentage Increase in Volume	Condition
Compound A[a]	**30 min. at 274 F. (134 C.)**	270	338	Brittle
	45 min. at 274 F. (134 C.)	222	278	Brittle
	60 min. at 274 F. (134 C.)	202	253	Brittle
	90 min. at 274 F. (134 C.)	176	220	Brittle
Compound B[a]	**30 min. at 274 F. (134 C.)**	302	372	Flexible
	45 min. at 274 F. (134 C.)	264	325	Flexible
	60 min. at 274 F. (134 C.)	235	289	Flexible
	90 min. at 274 F. (134 C.)	237	292	Flexible

[a] *Compositions*:

	Compound A, parts by weight	Compound B, parts by weight
Smoked sheets	100.0	100.0
Zinc oxide	10.0	10.0
Stearic acid	1.0	1.0
Phenyl-beta-naphthylamine	2.0	2.0
Diphenyl-guanidine	1.0	....
Tetramethyl-thiuram-monosulfide	....	2.6
Sulfur	5.0	0.4

Table I shows the changes in weight and volume that occurred when molded test specimens of these two compounds, 1 by 2 by 0.080 in. thick, were immersed one month in kerosine at 82 F. (28 C.) The test specimens were weighed before immersion; and after removal from the kerosine they were wiped dry with a cloth and weighed immediately. The percentage of increase in volume was calculated according to the formula:

$$\text{Percentage increase in volume} = \frac{\begin{array}{c}\text{Percentage increase in weight} \times \text{specific}\\ \text{gravity of sample}\end{array}}{\text{Specific gravity of kerosine}} \quad \ldots\ldots\ldots(1)$$

If the oil resistance of these two rubber compounds were judged only by the changes in weight and volume during immersion in kerosine, one would be forced to the conclusion that compound A is better than compound B. However, compound A lost practically all of its strength and toughness during immersion, and at the end of the test it was so tender that it could not be bent on itself without breaking. Compound B, on the other hand, retained its strength, extensibility, and toughness to a considerable degree, and is therefore a more serviceable compound for use in contact with kerosine. These data demonstrate clearly the fact that changes in weight and volume do not indicate the extent of other and more important physical changes that may take place when rubber is in contact with kerosine. In other words, dimensional changes do not parallel changes in other and more important physical properties.

Measurement of Dimensional Changes:

It is extremely difficult to measure accurately the changes in dimensions that occur in rubber during immersion in a swelling medium. Changes in length and width can be measured with a fair degree of accuracy if the specimen has undergone no distortion during immersion, but accurate measurement of thickness after immersion is quite impossible, due to the extreme softness of swollen rubber.

Perhaps the best method of measuring changes in volume during immersion is by water displacement. This is often referred to as the "Navy Method," and is used in U. S. Navy Department Tentative Specifications for non-metallic gasoline hose, No. 33 H 2 dated April 18, 1934. The necessary data may be quickly obtained with a Jolly or other suitable balance and the volume changes calculated according to the formula:

$$\text{Percentage increase in volume} = \frac{\text{Water displacement after test} - \text{Initial water displacement}}{\text{Initial water displacement}} \times 100 \quad . . (2)$$

Other observers, including the author, have determined change in volume by measuring change in weight and calculating the volume change according to Eq. 1. of this paper. This method of calculation requires the assumption that the volume of the rubber after immersion in the solvent is equal to its original volume plus the volume of liquid absorbed. It will give erroneous results if the volume of the rubber itself changes during immersion due to removal of extractable materials from the rubber by the solvent in which it is immersed. Erroneous results may also be obtained when the liquid in which the rubber is immersed contains two or more components having different specific gravities, because the two components may not be absorbed by the rubber to the same extent. Hence, when swelling tests are conducted in a mixed liquid, the specific gravity of the liquid absorbed may not be the same as the specific gravity of the swelling medium.

The Navy method (water displacement) is not open to these objections because it measures the percentage of increase in volume directly and does not involve the assumptions upon which the calculation of increase in volume from increase in weight is based.

Measurement of Weight Changes:

Test specimens should be wiped dry before weighing, and the time interval between the removal from the swelling medium and weighing should be kept constant and as short as possible in order to reduce to a minimum the possible error due to evaporation of the swelling medium.

Significance and Measure of Extraction and Sloughing:

Changes in weight and volume should never be taken as criteria of oil resistance without also taking into consideration the quantity and kinds of material extracted by the swelling medium. The presence of extractable material is indicated by changes in the appearance of the oil or solvent that is being used as the swelling medium. The quantity of extractable matter in natural rubber and in most rubber-like products is not great and is quite constant. However, compounding ingredients are often used which may be extracted in varying degrees by different swelling media.

The amount of matter extracted by a volatile solvent may be measured by evaporating the solvent to dryness and weighing the residue. There appears to be no satisfactory method of measuring the amount of material extracted by a non-volatile solvent.

Many rubber compounds will "slough off,"—that is, undergo a surface disintegration,—when immersed in oils, and deposit fine particles in the liquid. The contamination of the liquid in this manner is often very objectionable. For example, if a gasoline hose tube "sloughs off," the gasoline is discolored and the particles of rubber may clog screens, etc., causing no end of trouble and delay.

Accurate measurement of the degree of "sloughing off" is quite difficult. One may, of course, weigh the sample after drying out the liquid or weigh the residue that remains after evaporating a volatile liquid. But such tests fail to differentiate between solid particles that "slough off" and soluble material extracted from the compound. One method of differentiation between these two sources of contamination is to stir the test specimen gently in the liquid after completion of the swelling test, strain the liquid, and weigh the dried residue collected on the screen. However, it is believed that the most satisfactory test is to examine the sample carefully during and at the completion of the test and to determine the extent of surface disintegration by wiping it with a clean cloth or by scraping lightly with a penknife.

Significance of Changes in Physical Properties:

No rubber compound may properly be called oil resistant unless its strength, resilience, toughness, etc., are maintained in a high degree during contact with the oils to which it will be exposed in service.

In considering methods for determining changes in these physical properties during contact with oils, it is necessary to differentiate sharply between two types of tests. If the product is subjected to abrasive wear or to other mechanical stresses while immersed in or still saturated with the swelling agent, it seems obvious that tests for changes in physical properties should be conducted immediately after removal of the specimen from the testing liquid. Physical tests conducted at this time may be called "deterioration tests." On the other hand, if the conditions of service are such that the product will be subjected to severe mechanical strain some time after removal from contact with the solvent, it is then desirable that physical tests be conducted after a period of rest following removal from the swelling medium. Tests conducted at this time may be called "recovery tests." Obviously, the rest period should be sufficient to permit volatilization of a considerable portion of the swelling agent.

Most rubber or rubber-like products called upon to resist the action of oils and solvents are subjected to mechanical strains during contact with the oil. In testing for this condition of service, deterioration tests should be applied rather than recovery tests. On the other hand, certain products are used under conditions of service where recovery tests should be applied rather than deterioration tests. An example is tank-loading gasoline hose which is used intermittently and is subjected to the most severe mechanical strains, not while in use, but rather after having been used and having had an opportunity to "recover," that is, to lose by volatilization a considerable portion of the gasoline it has absorbed.

The time interval between the removal of the test specimen from the swelling agent and making the test does not necessarily classify the test as "recovery" or "deterioration." For example, a tension test made on a test specimen one hour after removal from gasoline would be classed as a "recovery test," whereas the same test made one hour after removal from lubricating oil would be classed as a "deterioration test," because in the first instance the test specimen has recovered somewhat due to the volatilization of the gasoline, whereas in the second case no appreciable amount of the lubricating oil has evaporated.

Measurement of Changes in Physical Properties:

There are no testing machines capable of measuring accurately the tensile strength, elongation at break, elastic modulus, tear resistance, and abrasion resistance of a rubber compound while it is swollen and wet with a solvent. Consequently, when one wishes to determine the changes in these properties during immersion in a swelling agent, he must rely on manual tests with the aid of such simple tools as the fingernail, a blunt screw driver, and a pocketknife. Such tests are, of course, incapable of numerical expression, but in the hands of an experienced operator they may be developed to a surprisingly high degree of accuracy. Tensile strength and elongation are estimated by stretching the sample until it breaks. The stiffness or elastic modulus is determined by stretching the swollen sample and making a mental note of the approximate force required to stretch it to a given extent as compared with the force required before immersion. The tear resistance can be determined with the fingernail or with a screw driver, and should be correlated mentally with the grain effect which is aggravated in swollen rubber. Abrasion resistance can be judged by scraping with a penknife.

If the conditions of service to which the product will be subjected are such that a recovery test is significant, it then becomes practical to apply the conventional machine tests for tensile strength, elongation, modulus, and tear resistance. It must be borne in mind, however, that the conventional testing machine is not at all well suited for the determination of physical properties of badly deteriorated samples. Hence, if recovery tests are to be of value, the immersion test must be stopped at a moderate degree of deterioration. It is believed that any machine tests for tensile strength are of little value on samples having a tensile strength of less than 500 lb. per sq. in. Below this point the manual tests are more significant.

Machine abrasion tests are of no value whatever on a sample that has been exposed to swelling agents, because the unavoidable residue of swelling agent in the sample lubricates the abrasive track.

FACTORS AFFECTING OIL RESISTANCE TESTS

Effect of Temperature:

All rubber and rubber-like compounds are more rapidly attacked by oils at elevated temperatures than at normal temperatures. It is frequently desirable to conduct tests at elevated temperatures either for the purpose of duplicating service conditions or to accelerate the test so that the testing engineer may forecast the performance of the compound in the shortest possible time. The practicability of heat-accelerated tests depends upon the type of composition being tested and upon the solvent. Obviously, compositions that will be subjected in service only to normal temperatures should not be given heat-accelerated tests if their physical properties are seriously affected by elevated temperatures. Neither is it practical to use the heat-accelerated test when studying the effects of volatile or of flammable solvents.

It is often possible to select a non-volatile solvent or blend of solvents that may be used at elevated temperatures to produce effects comparable to the effect produced by immersion in volatile solvents over a long period of time at normal temperatures. Obviously, such a test cannot properly be specified until the testing engineer has accumulated sufficient data to prove that the liquid chosen for test purposes at elevated temperatures produces effects comparable to those produced by the liquids that may be encountered in service.

The weight of experimental evidence indicates that heat acceleration is a practical means of forecasting the performance of most compositions in solvents at normal temperatures.

Effect of Time:

The time required to deteriorate rubber in contact with oils is influenced by the size and shape of the test specimen, by the temperature of the test, and by the rate of diffusion of the swelling medium.

Vulcanized rubber usually swells rapidly during the early period of oil immersion and then falls off to a slow but gradual increment, which increment may be responsible for a considerable part of the total swelling.[1]

In any test for the oil resistance of rubber the time element is an important factor, and the investigator should avoid conclusions based on tests that are not run for a sufficient length of time to insure that no further appreciable decrease in physical properties will occur.

Effect of Size and Shape of Test Specimen:

Differences obtained in the results with test specimens of various shapes and sizes are greatest during the early periods of immersion, which is to be expected because the rate and degree of absorption and diffusion of the swelling agent are important factors. It is obvious that a thin test specimen will be more seriously attacked by oil in a shorter period of time than a thick specimen, which emphasizes the fact that test specimens of the same shape and size should be used to obtain comparable results.

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[1] J. R. Scott, "The Swelling of Vulcanized Rubber in Liquids," *Transactions*, Inst. Rubber Industry (London), Vol. V, No. 2 (1929).

Effect of Aging:

The effect of aging is to reduce the oil resistance unless the rubber is so compounded as to increase in stiffness with aging, in which case the oil resistance may be slightly improved. Aside from the effect of aging on oil resistance, it is obviously necessary, from a purely commercial point of view, to have a good aging compound to prevent deterioration both before and during service in oil. Many so-called oil-resisting rubber compositions used in the past were notoriously poor agers, but fortunately the modern trend of compounding rubber for oil resistance has been also to improve the aging qualities.

The testing of freshly vulcanized rubber compositions for their physical properties is conceded to be poor practice and is particularly to be discouraged in testing for oil resistance. Test specimens should be allowed several days' rest before immersion, in order to reach an equilibrium approximating that of the condition during service.

Influence of External Mechanical Forces:

The oil resistance of rubber and rubber-like products is influenced by the amount and type of mechanical forces applied during immersion. These forces may be static or dynamic, and the composition may be subjected to compression or extension, or both. When the swelling of rubber is restricted by mechanical pressure, the amount of oil absorbed is naturally reduced. When the composition is in motion or in a strained condition, the rate of oil diffusion and of absorption increases.

The scope of this paper is not broad enough to discuss the effect of these mechanical forces in a manner worthy of their importance. They are mentioned only in order to suggest to the testing engineer that in establishing his performance tests, he should use such test methods as will give results that can be interpreted in terms of oil resistance under service conditions.

Effect of Quality of Swelling Media:

Not only do different oils and solvents affect rubber and rubber-like compounds in a different manner and to a different degree, but variations in the quality of a given type of oil or solvent often have a marked effect on the test results, and this is particularly true of petroleum products, as was shown by Hayden and Krismann[1] in a study of the effect of immersing a DuPrene compound in various types of crude and refined petroleum products. In this article it was shown that a Pennsylvania crude oil, which consists largely of saturated aliphatic hydrocarbons, caused the DuPrene stock to swell much less than the Midcontinent crude oils, which are somewhat unsaturated. Coastal crude oil, which is still more unsaturated and is composed largely of aromatic (naphthenic) hydrocarbons, caused more swelling of the DuPrene compound than either the Midcontinent or Pennsylvania crude oils. Variable results were also shown with motor fuels and lubricants from different sources. It is therefore very important to use solvents of known source and quality when testing the oil resistance of rubber and rubber-like compositions.

SUMMARY

The technique of testing the oil resistance of rubber and rubber-like materials is less accurate than that for many other qualities. It is impossible to obtain exact measurements of the most significant changes that occur during immersion in a swelling medium. The problems facing testing engineers are to develop observation and manual tests which will supplement machine tests, to develop accelerated tests that can be correlated with service conditions, and to interpret test results in values which will demonstrate the performance of these products in oil or solvent service.

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[1] O. M. Hayden and E. H. Krismann, "Effect of Oils and Chemicals on DuPrene Compounds," *Industrial and Engineering Chemistry*, Vol. 25, p. 1219 (1933).

DISCUSSION

MR. G. C. MAASSEN[1] (*presented in written form*).—Referring to Mr. Hayden's paper, there is a suggestion I should like to make in reference to the measurement of weight changes. It is believed that when rubber specimens have been immersed in solvents, it is inadvisable to wipe dry before weighing, especially when the solvent causes sloughing. By wiping, it is evident that all conditions being equal, the sample that sloughs off on the surface will have enough material wiped off to favor it in the swelling test.

When volatile solvents are used, the rate of evaporation is usually great enough so that the surface is as dry in about a minute's time as any wiping may make it. When non-volatile solvents are used, it is recommended that the sample be rinsed in a volatile solvent in which the non-volatile liquid is soluble. Thus by eliminating the wiping action, the material which sloughs off and becomes cheesy on the surface has no advantage over the material which remains firm throughout the test.

MR. O. M. HAYDEN.[2]—Wiping or blotting a surface that is badly deteriorated from contact with oil will undoubtedly affect the results. A great deal of care must be exercised in wiping dry a swollen test specimen in order not to remove any of the surface. Significant data are usually obtained before serious surface disintegration occurs by taking weight change measurements at frequent intervals during the tests. Mr. Maassen's suggestion is a valuable contribution and is worthy of serious consideration.

MR. ARTHUR W. CARPENTER.[3]—In discussing the factors affecting oil resistance tests, the author summarizes known principles governing the action of solvents on rubber which have been demonstrated by many workers in extensive research studies. Upon consideration, it is noted that these principles deal with the results from contact of rubber and solvent rather than with the fundamental causes and mechanism of the ensuing action. In the present paper, limitations as to both time and space doubtless compelled the author to forego any discussion of the various theories of the action of solvents on rubber. It would seem, however, that the development of satisfactory test methods for evaluating oil resistance would require precise understanding not only of the effects which oil produces in rubber but also of the causes and mechanism of the action. It is conceivable that such knowledge would entirely change the mode of attack of the test problem.

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Though much work has been done and valuable knowledge gained concerning the general effects of oils on rubber, it is amazing how little is known regarding precisely what happens internally when the two materials come together. In 1907, Spence[1] gave a theory of the behavior of unvulcanized rubber toward solvents while discussing the protein constituent of crude rubber. Later, Posnjak,[2] treating the swelling of rubber as a problem in colloid chemistry, measured swelling pressures using a series of solvents and developed some mathematical relationships. He concluded that velocity of swelling is a reaction of the first order and that, on the whole, swelling appears to be a capillary phenomenon. Many writers have since discussed the effects of solvents on rubber. The paper by J. R. Scott, referred to by the author, is perhaps the most comprehensive recent treatment of the problem of swelling and deals entirely with vulcanized rubber. Scott states in his introduction, however, that the mechanism of the absorption of liquid and the structure of swollen rubber gels are not discussed. Naunton, Jones and Smith[3] have presented a theory of the action of oil on rubber which treats the problem largely from a structural viewpoint. It would seem that the time is ripe for more fundamental research. The diffusion of the liquid into the rubber should be carefully investigated and the laws governing it established. Perhaps an osmotic study would be helpful. Such work should be of value in clarifying the test problem as well as possibly in indicating methods for development of rubber compounds having better oil resistance than those used at present.

The author has pointed out the difficulty of testing the tensile strength of rubber specimens after they have been immersed in oil. He indicates the lack of availability of suitable testing equipment, mentioning that the conventional equipment cannot be used successfully for rubber-like materials that have tensile strengths below possibly 500 lb. per sq. in. This is entirely true under ordinary conditions. However, there are now available low-capacity tension testing machines suitable for use with rubber samples requiring total loads for rupture as low as 50 g. Machines of this type are in use for testing thin specimens, such as rubber tape. Therefore, while we may not use the conventional 150-lb. capacity tension testing machine when we are dealing with total loads of a few ounces, it is possible by selecting the proper equipment to carry out tension tests on very weak rubber specimens with a fair degree of success. A similar situation exists in connection with the problem of holding the samples in the grips. Of course, after the test specimens are immersed in oil, they are slippery and are difficult to hold in clamps of the usual type. However, ring specimens have been successfully used and by employing properly designed wedge jaws, it is possible to hold swelled dumb-bell specimens quite well. I cannot agree that the use of tension tests as a measure of deterioration of rubber after immersion in solvents is as definitely out of the question as the author indicates.

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[1] D. Spence, "Distribution of the Protein in Para Rubber," *Quarterly Journal*, Inst. Commercial Research in Tropics, Liverpool, 1907, pp. 47–60; reprinted in *India Rubber Journal*, Vol. 34, p. 766, and Vol. 35, p. 23 (1907).

[2] E. Posnjak, "Über den Quellungsdruck," *Kolloidchemische Beihefte*, Vol. 3, p. 417 (1912).

[3] W. J. S. Naunton, Maldwyn Jones, and W. F. Smith, "Oil Resisting Rubber," *Transactions*, Inst. Rubber Industry, Vol. 9, No. 3, October, 1933, p. 169.

It is unfortunate that the author was handicapped in presenting this paper by having to deal with a subject so broad that much specific test data could not be given. I am sure that we would all like to see rather more extensive data on this subject. Since the Society's Committee D-11 on Rubber Products now has a subcommittee which is actively at work under the chairmanship of the author on the problem of testing oil resistance, doubtless, in subsequent communications, more specific test information will be presented. The subject is so broad that detailed treatment must necessarily be restricted to limited phases. It seems therefore that the author chose wisely in presenting in this first paper a general view of the problem. I venture to hope that we may be favored with future papers dealing in detail with the separate factors involved in the testing of rubber for oil resistance.

MR. HAYDEN (*by letter*).—Undoubtedly an understanding of the cause and mechanism of the action of solvents on rubber would enable us to devise better methods of evaluating oil resistance and certainly it would contribute greatly to the development of solvent-resisting rubber compositions. The theory presented by Messrs. Naunton, Jones and Smith should stimulate fundamental research on the subject.

I do not question the possibility of obtaining tension test results on very weak rubber specimens by the use of low-capacity tension testing machines. However, I doubt whether differences between tensile strength results of less than 500 lb. per sq. in., on rubber that has been weakened by immersion in solvents, are very significant. Surface defects, dispersion of compounding ingredients and other factors have a profound influence on the amount of work required to rupture degraded rubber. It has been observed that there is a tendency for tension test results to level out when the tensile strength of rubber compositions falls below 500 lb. per sq. in.

THE TESTING OF RAW MATERIALS FOR RUBBER COMPOUNDS

By B. S. Taylor[1]

Synopsis

Raw materials used in rubber manufacture must be of such a nature that they will perform properly their respective functions in the presence of the standard vulcanized rubber system of rubber-sulfur-zinc oxide-accelerator. After vulcanization they must remain unchanged and not bring about changes in the rubber.

The critical points to consider in evaluating a raw material for rubber are:

1. Ease of incorporation into rubber batches.
2. Effect on rate of vulcanization and aging.
3. Ability to reinforce or soften rubber compounds.
4. Effect in imparting special physical properties.
5. Properties involving human factors—color, odor, toxicity, consistency.

The physical and chemical properties used to evaluate the two most important rubber compounding ingredients, zinc oxide and carbon black, are thoroughly described. The information obtained from the study of these materials is applied to the testing of all rubber compounding materials that are classed as pigments.

Tests are run for two equally important reasons: To determine desirable properties and to detect traces of impurities. A number of important impurities are described and their undesirability explained.

The raw materials ordinarily used for the preparation of rubber compounds may be considered in three classes. The first of these consists of crude rubber or rubber hydrocarbon in its various forms. The second class includes (*a*) vulcanizing agents such as sulfur, (*b*) accelerators of vulcanization, (*c*) activators or materials which may be designated as vulcanization aids and (*d*) antioxidants which act to preserve the vulcanized rubber mixture. Finally, there is that large group of materials often referred to in the rubber industry as pigments though including many substances having no relation to coloring effect. These may be liquids, waxes or resins as well as dry powders. The solid materials may be coarse or extremely finely divided. Some are chemically or physically active and others are entirely inert. The materials of this class are by far the most numerous and at present are responsible for the largest part of the raw materials testing in the rubber industry.

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[1] Manager, Raw Materials Inspection and Development, The B. F. Goodrich Co., Akron, Ohio.

Crude rubber, itself, is extremely difficult to evaluate by any test procedure. Like most substances of vegetable origin, it is obtained in many different forms, and varies with the climate and location of production as well as with many other factors. The nature of the material renders satisfactory sampling nearly impossible, and experience has demonstrated that better control results can be obtained at less cost through inspection and classification by experts in rubber grading than by chemical or physical tests. The materials of the second class, however, are usually evaluated by test and some of the details are discussed herein since it is necessary to have a rather thorough knowledge of the part played by them in order to understand fully many of the specifications written for materials of the third class, namely, pigments intended for use in rubber compounds. For the most part, this discussion deals with dry powders and the materials classed as pigments. The tests now employed on the two most important ones are described in detail and the knowledge gained from these materials are applied to the general class of rubber pigments.

Usually all pigments must conform to certain types of requirements which determine the nature of the tests used. A simple rubber mixture usually contains rubber, sulfur, a small amount of zinc oxide and accelerator, sometimes fatty acid and at times other metallic oxides as vulcanization aids. Such a mixture containing no dry powder as a diluent or loading material is known as pure gum. These pure gum mixtures are of value only in the manufacture of a limited number of kinds of articles. Usually, pigments are added to produce changes in physical properties desired for some particular use to which the article is to be put.[1] The specifications drawn for the various pigments indicate to what degree these materials affect the physical properties of the cured rubber. Also incorporated in these specifications are the maximum limits for certain impurities. Consideration must always be given in judging raw materials for rubber manufacture that after vulcanization there shall occur no further change in the rubber due to the presence of impurities and that the compound shall retain the properties imparted to it by the particular pigment present. It may be said in general that chemical tests of a material are used to determine the presence of impurities, often in very small amounts, while physical tests of rubber mixes containing the ingredient are used to demonstrate the degree to which the material fulfills its purpose in rubber compounding.

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[1] R. H. Seeds, "Compounding: A Discussion of the Fillers, Reinforcing Agents and Softeners Used in the Compounding of Rubber," Symposium on Rubber, Cleveland Regional Meeting, Am. Soc. Testing Mats., p. 46 (1932).

In evaluating a raw material, five critical points must be considered:

1. Ease of incorporation into rubber batches.
2. Effect on rate of vulcanization and aging.
3. Ability to reinforce or to soften rubber compounds.
4. Effect in imparting special physical properties.
5. Properties involving human factors—color, odor, toxicity, consistency.

The first qualification, ease of incorporation, is difficult to describe in a specification in such a way as to make the requirements understandable to both manufacturer and user of a pigment. It is a consideration of the utmost importance to the rubber manufacturer because it vitally affects the cost of batch preparation. Usually the requirement of uniform processing quality must be covered by stating in a specification that the material must be equal in this respect to an agreed-upon standard sample. Theoretically such a clause could be made to include all essential points of any specification. However, it is more fair to a supplier to include in purchase specifications as many definite test requirements as possible and to employ this general clause only to cover some quality which might otherwise be overlooked. In actual practice, factory batches are mixed for the determination of processing qualities, until it is certain that these do not vary with successive shipments. The sources of the materials are then approved.

The second quality, the effect on rate of vulcanization and aging, is easily determined by incorporating the pigment in high percentages in a standard rubber batch and examining the changes in stress-strain data obtained by testing a series of vulcanized specimens of the batch. This type of test is well known and in very general use. By means of it, the effect of pigments on the vulcanization aids present in the ordinary rubber batch may be appraised.

The third quality, ability to reinforce or soften rubber compounds, is measured by the same physical tests as rate of cure except that in this case the emphasis is on the physical properties themselves rather than in their rate of change with time. The measurements of stress-strain relation and tensile strength by standard rubber tension testing machines are accurate enough to show the difference between pigments of various classes, and to determine the uniformity of particle size and to some extent particle shape. The relative effectiveness of softeners may be judged from plasticity tests on the original batch, by measuring chemical properties of the softener, and by testing the cured rubber for stress-strain relationship, tensile strength and ultimate elongation.

The fourth quality, effect in imparting special physical properties, involves different testing for each kind of material considered. Substances such as cork, graphite, shellac, aluminum powder, asbestos and wood flour are usually compounded in rubber which is subsequently vulcanized and submitted to tests such as hardness, set, transverse strength and others which evalute those qualities in a pigment that render it of value for its special uses in rubber.

Those properties of raw materials involving human factors, odor, toxicity and consistency, are often a matter of merely qualitative testing. This is not true in the case of color which again involves compounding into a particular rubber batch and making a comparison with standard pigment color. The toxicity of raw materials and odors due to impurities may render a raw material unfit for use in rubber. Consistency of oils and softeners plays an important part in the ease of working these materials into rubber on the mill and, from a purely personal standpoint, in the ease of handling of the ingredient before putting it into rubber. Many factors affecting personnel relations must be considered in evaluating a raw material for use. Quite often if a pigment flies badly when being mixed, this is just as severe an indictment from the standpoint of manufacturing operations as poor quality in rubber tests.

EVALUATION OF ZINC OXIDE FOR USE IN RUBBER

Turning now to the two most important rubber pigments, the criteria outlined above will be applied to zinc oxide and channel gas black. Zinc oxide is used in rubber in amounts varying from 1 to 70 per cent of the batch. There are very few cured rubber articles that do not contain this pigment. Zinc oxide is used in large percentages for reinforcement or in small amounts for activation of organic accelerators of either acid or basic types. It is also of extreme importance for white or light-colored goods. Suppliers of the material produce a variety of pigments under the name "zinc oxide," all of which contain the substance ZnO but in addition, certain impurities. The governing chemical factor in its use in large proportions seems to be not the oxide itself but rather the impurities in it or on its surface. The other factor of importance is that of particle size, but the significance of this property has often been wrongly estimated because of the effect of impurities present. It is well known that some batches containing large quantities of zinc oxide are difficult to mix on a mill. The reason for this difficulty could not be determined with accuracy until recently because of the impossibility of controlling all of the factors involved in roll mill mixing. With the increasing use of the

Banbury mixer for preparing masterbatches, differences were promptly found between zinc oxides. The pigment is much used in masterbatch form—that is, it is first mixed into rubber in a high concentration and this mixture is subsequently weighed into the final batch and serves to introduce the zinc oxide. This method assures accuracy both in weighing and in distribution of pigment in the ultimate compound. Differences in dispersibility are readily demonstrated in this masterbatch as Stutz, Haslam and Silver have recently shown.[1] Depew also has published a résumé on "Zinc Oxide in Rubber" which includes a reference to many of the requirements for satisfactory material.[2] For the present work, a large number of zinc oxides have been studied, thirteen of which are defined in the tables, lettered A to P.

TABLE I.—EFFECT OF ACIDITY, PARTICLE SIZE AND TREATMENT OF SURFACE.
Mineral Acid calculated as per cent H_2SO_4.

Code	Process	Lead, per cent	Acid, per cent	Micro-Examination					Surface Treatment
				Size, μ			Percentage by Weight Below 1 μ	Banbury Dispersion	
				Max.	Min.	Avg.			
EFFECT OF ACIDITY									
A................	American	0.05	0.23	4	0.1	0.6	85	Poor	None
B................	American	0.06	0.08	4	0.1	0.6	85	Good	None
EFFECT OF PARTICLE SIZE									
C................	French	0.02	0.002	4	0.1	0.6	75	Excellent	None
D................	French	0.01	0.004	2	0.1	0.4	95	Only fair	None
EFFECT OF TREATMENT OF SURFACE									
D................	French	0.01	0.004	2	0.1	0.4	95	Only fair	None
E................	French	0.01	0.01	2	0.1	0.4	95	Excellent	0.3 per cent fatty acid
F................	French	0.07	0.004	2	0.1	0.25	95	Poor	None
G................	French	0.05	0.01	2	0.1	0.3	99	Excellent	0.35 per cent fatty acid

The principal factors influencing mixing of zinc oxide are:

1. Mineral acidity of the oxide.
2. Particle size.
3. Treatment of surface.

The effect of acidity due to mineral acid, of particle size, and of surface treatment with fatty acid is shown in Table I.

The extent to which a zinc oxide affects the rate of vulcanization depends upon the concentration of the oxide in the compound.

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[1] G. F. A. Stutz, G. S. Haslam and B. R. Silver, "Zinc Oxides," *The Rubber Age*, June 10, 1932; June 25, 1932; July 10, 1932.

[2] H. A. Depew, "Zinc Oxide in Rubber," *Industrial and Engineering Chemistry*, Vol. 25, p. 532 (1933).

Small amounts of oxide up to 5 parts by weight to 100 lb. of rubber in the recipe produce satisfactory cures regardless of impurities or variations in particle size. When higher concentrations of the material are used to produce special properties, the extent to which the particle size or impurities affect the rate or state of cure, varies directly with the percentages used. The test results which are given to show the effect of impurities have been secured using recipes containing about 20 volumes of zinc oxide to 100 volumes of rubber (weight ratio 112 to 93). The principal factors of which the influence on rate of cure has been studied, using a high concentration of zinc oxide, are:

1. Acidity of the oxide causing it to act as a retarder for accelerators. The effect is most marked in the case of the aldehyde amine accelerators.

TABLE II.—EFFECT OF ACIDITY ON CURE.

Analysis:	OXIDE CODE H	OXIDE CODE J
Lead, per cent	0.32 as Pb	0.30
Acid, per cent	0.35 as H_2SO_4	0.09
Microexamination, μ:		
Max	4	4
Min	0.1	0.1
Avg	0.6	0.6
Percentage by weight below 1 μ	85	75

RUBBER COMPOUNDING TESTS

Recipe No. 234 (butyraldehyde aniline):

CURE	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.
10 min. at 275 F	1700	675	450	2800	690	800
30 min. at 275 F	3400	695	900	3700	645	1300
40 min. at 275 F	3400	675	1000	3800	625	1400
110 min. at 275 F	3500	640	1250	3700	610	1400

Recipe No. 224 (hexamethylene tetramine):

CURE	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.
10 min. at 307 F	2450	630	900	2650	590	1150
15 min. at 307 F	3200	675	1050	3000	600	1250
25 min. at 307 F	3450	630	1300	3100	590	1400
40 min. at 307 F	3300	600	1450	3150	580	1550
55 min. at 307 F	2675	550	1525	2150	480	1650

2. The presence of lead oxide which functions as an activator for all basic accelerators and some acid accelerators such as mercapto benzothiazol.

TABLE III.—ACTIVATION EFFECT OF LEAD.

Analysis:	OXIDE CODE J	OXIDE CODE K
Lead, per cent	0.30 as Pb	0.06
Acid, per cent	0.09 as H_2SO_4	0.08
Microexamination, μ:		
Max	4	4
Min	0.1	0.1
Avg	0.6	0.6
Percentage by weight below 1 μ	75	85

RUBBER COMPOUNDING TESTS

Recipe No. 234 (butyraldehyde aniline):

CURE	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.
10 min. at 275 F	2800	690	800	2000	690	600
30 min. at 275 F	3700	645	1300	3600	670	1100
40 min. at 275 F	3800	625	1400	3600	650	1250
110 min. at 275 F	3700	610	1400	3600	630	1350

TABLE IV.—ACTIVATION EFFECT OF LEAD.

Analysis:	OXIDE CODE L	OXIDE CODE M
Lead, per cent	0.6 as Pb	0.05
Acid, per cent	Neutral	0.001
Microexamination, μ:		
Max	0.5	0.5
Min	0.1	0.1
Avg	0.2	0.2
Percentage by weight below 1 μ	100	100

RUBBER COMPOUNDING TESTS

Recipe No. 234 (butyraldehyde aniline):

CURE	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.	TENSILE STRENGTH, LB. PER SQ. IN.	ELONGATION, PER CENT	LOAD AT 400 PER CENT ELONGATION, LB. PER SQ. IN.
10 min. at 275 F	3700	725	750	2600	705	500
30 min. at 275 F	4050	685	1000	3800	700	850
40 min. at 275 F	4050	685	1000	4000	700	900
110 min. at 275 F	2900	540	1400	4000	640	1400

3. The presence of lead compounds which affects the cure of rubber with thiuram and dithio carbamate accelerators.

4. Particle size which affects cure very little.

These facts are brought out in the tests shown in Tables II, III and IV. It will be noted in Table II that the difference between the oxides is that of acidity, that the effect of this acidity is very marked on rubber stock accelerated with an aldehyde amine of the type of butyl-aldehyde-aniline and not noticeable when accelerated with hexamethylene tetramine. In Table III the difference between the oxides is that of lead content, and the oxide carrying a small amount of litharge cures more rapidly than that which contains only a trace of lead. A similar comparison is made in Table IV using very finely

Table V.—Retarding Effect of Lead on Thiurams.

Same Oxides as Table IV.

Oxide Code L (0.6 per cent lead) Oxide Code M (0.05 per cent lead)

Rubber Compounding Tests

Recipe No. 109-BT (*tetra methyl thiuram disulfide*):

Cure	Tensile Strength, lb. per sq. in.	Elongation, per cent	Load at 500 per cent Elongation, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation, per cent	Load at 500 per cent Elongation, lb. per sq. in.
10 min. at 275 F.........	1000	790	200	3600	700	1550
20 min. at 275 F.........	2450	750	1100	3600	680	1525
30 min. at 275 F.........	3050	720	1100	3600	680	1625
40 min. at 275 F.........	2810	690	1150	3500	690	1500

Table VI.—Effect of Particle Size.

Analysis:	Oxide Code F	Oxide Code P
Lead, per cent..........................	0.07 as Pb	0.02
Acid, per cent..........................	0.004 as H_2SO_4	0.002
Microexamination, μ:		
Max..................................	2	5
Min..................................	0.1	0.1
Avg..................................	0.25	0.6
Percentage by weight below 1 μ...........	95	75

Rubber Compounding Tests

Recipe No. 234 (*butyraldehyde aniline*):

Cure	Tensile Strength, lb. per sq. in.	Elongation, per cent	Load at 400 per cent Elongation, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation, per cent	Load at 400 per cent Elongation, lb. per sq. in.
10 min. at 275 F.........	3150	720	650	3350	680	850
30 min. at 275 F.........	4100	710	1000	3900	655	1200
40 min. at 275 F.........	4150	700	1200	3900	625	1300
55 min. at 275 F.........	3700	650	1450	3550	610	1300

divided zinc oxide, and again the lead content activates the rubber compound accelerated with a basic accelerator. Oxide L cures faster initially than oxide M and overcures more. Table V shows the retarding effect of lead in zinc oxide on cures with a thiuram disulfide accelerator. In Table VI the difference between the oxides is that of particle size. These two oxides have the same rate of cure although oxide F is considerably finer than oxide P. The recipes used in testing zinc oxide are as follows:

Recipe No. 109-BT:		*Recipe No. 224:*		*Recipe No. 234:*	
Rubber	100.00 parts by weight	Rubber	466.50	Rubber	46.50
Tetramethyl thiuram disulfide	0.25 parts by weight	Sulfur	46.25	Sulfur	1.50
Sulfur	3.00 parts by weight	Hexam	2.25	808 Accelerator	0.20
Zinc oxide	100.00 parts by weight	Zinc oxide	635.00	Zinc oxide	56.30
		Approximately 23 volumes per 100 volumes of rubber		Exactly 20 volumes per 100 volumes of rubber	

The evaluation of zinc oxide as regards the last two general requirements, those of special physical properties and personal factors, is covered by the whole specification which carefully controls the pigment. Although zinc salts are considered to be poisonous, zinc oxide as a material to be handled by human beings is ordinarily not considered toxic.

A specification for the ideal zinc oxide for general use includes the following requirements:

Lead, per cent, max	0.08
Sulfide metal such as cadmium, bismuth, etc., per cent, max	0.10
Acidity calculated as H_2SO_4, per cent, max	0.01

In addition to these requirements, testing of the oxide in a standard recipe accelerated with hexamethylene tetramine should be required since curing with this accelerator brings out certain factors not mentioned elsewhere in the specification, such as color after vulcanization and particle size. The latter is fairly well indicated by the load at 400 per cent, the tensile strength and tear resistance. A valuable check on the chemical analysis is also afforded in that this recipe will demonstrate the presence of lead by both rate of cure and color. The specification for zinc oxide includes a number of other requirements such as color, feel, and fineness of the raw material, but these are part of every specification for rubber compounding ingredients.

EVALUATION OF CARBON BLACK

Carbon black, another pigment of great importance in rubber manufacture, is used in all automobile tire treads and in practically every kind of rubber used for abrasion resistance. This material aptly illustrates the necessity for a close scrutiny of laboratory behavior before release for factory production. The two major problems in evaluating channel gas black for use in rubber are the same as those for zinc oxide: namely, effect on accelerators and ease of incorporation into rubber. The effect on accelerators is always tested by mixing a small rubber batch and determining the rate of cure. No chemical properties readily determined have as yet been found practical by rubber manufacturers as a means for evaluating carbon black for rate of cure, though several papers have been published by individuals of the carbon black industry on the use of the high-temperature volatile loss determination as a control test in determining the rate of cure of carbon black in rubber.[1] This method may be satisfactory to the pigment manufacturer for it is particularly applicable to the control of gas black from a given manufacturing unit. It is not of value, however, as a laboratory measurement for carbon blacks where a comparison of the products of a number of plants is necessary. For use with basic accelerators such as the aldehyde amines or guanidines, a much closer scrutiny of rate of cure is necessary than for acidic accelerators of the mercapto benzo thiazole or thiuram type. For example, two channel gas blacks, differing from one another when cured in a recipe containing diphenyl guanidine by as much as 15 min. in time necessary to reach the optimum state of cure at 290 F. (143 C.), could nevertheless be used with mercapto benzo thiazole interchangeably.

Although the function of carbon black in affecting rate of cure can be readily evaluated in the laboratory, measuring the ease of dispersion in rubber presents much more difficulty. Much time has been spent in attempting to develop a laboratory method of dispersing black using the 12-in. roller mill, which is the usual laboratory mixing equipment. Because of the tremendous amount of mechanical work done on the rubber batch by the small mill, small differences in power consumption cannot be accurately measured. Without this information, it is practically impossible to differentiate between carbon blacks which disperse easily or with great difficulty. Differences in ease of incorporation into rubber can be readily demonstrated using a factory Banbury mixer, and a few manufacturers and consumers have installed laboratory Banbury mixers which are small replicas of the large machines. These little Banbury mixers have not been found altogether satisfactory for the purpose of testing dispersibility of carbon black except under very special conditions. Development work has been in progress on the use of the Schiller mixer for the evaluation of the dispersion factor of carbon black and very interesting results have been secured by Schoenfeld and Allen.[1] When this method is finally worked out on a practical basis as a method of test, it should be of great value both to the producer and consumer of carbon black.

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[1] C. R. Johnson, "Carbon Black I, A Study of Its Volatile Constituents," *Rubber Chemistry and Technology*, Vol. 1, p. 465; "Carbon Black II, The Role of Oxygen," *Rubber Chemistry and Technology*, Vol. 3, p. 122.

Determining the amount of grit in channel gas black has been responsible for one of the most discussed phases of carbon-black testing. The specification requirements of the various companies all differ with respect to the amount of grit allowable. Carbon black is tested for grit by the wet screening method; an 80-mesh screen was the standard testing equipment until recently when one consumer instituted the use of a 325-mesh screen. The presence of grit in the amounts retained on an 80-mesh screen has not yet been proved to be harmful, although several papers[2,3] have been written on both sides of this controversy.

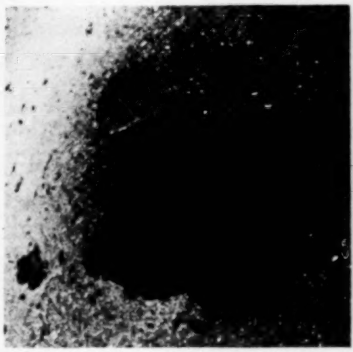
FIG. 1.—Showing So-called "Hard Agglomerate."

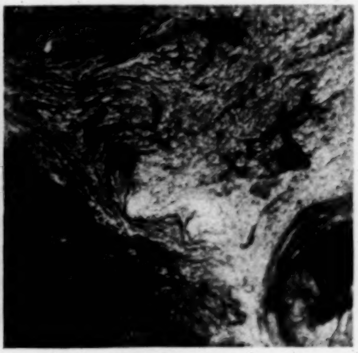
FIG. 2.—Showing Good Channel Gas Black Going into Rubber.

Carbon black is also tested for acetone extract, benzol extract and heating loss at 105 C. (221 F.). The extracts are of value in determining the amount of non-carbon material which may bleed from an underlying layer of carbon black compound to the surface of a white rubber cover and cause discoloration. The heating loss determines the amount of moisture and hydrocarbons volatile at the temperature of the test. Carbon black with a heating loss greater than 3 per cent is undesirable because there is a possibility that rubber stock containing a considerable portion of this type of black will be porous after cure.

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[1] E. K. Schoenfeld and R. P. Allen, "Dispersibility of Gas Black," *Industrial and Engineering Chemistry* Vol. 25, October, 1933, p. 1102.

[2] J. F. Street, "Grit in Carbon Black," *Industrial and Engineering Chemistry*, Vol. 24, p. 559 (1932).

[3] D. W. Kearsley and C. R. Park, "Effect of Grit upon the Stress and Strain Properties of a Carbon Black Stock," *Rubber Chemistry and Technology*, Vol. 4, p. 404.

PIGMENTS—DRY POWDERS

It is possible to outline certain general requirements for pigments in general, applying some of the information obtained on gas black and zinc oxide in the evaluation of other materials. Among dry powders, calcium carbonate, either dry ground or chemically precipitated, calcium sulfate, clay, barium sulfate, barium carbonate, slate flour and a wide variety of special materials have been widely used by the rubber industry. The type of chemical compound used as a pigment for either loading or reinforcing is unimportant, provided the material is not chemically reactive under the conditions of vulcanization and afterward. The heating loss for 2 hr. at 105 C. is an important test for determination of moisture. The ash or ignition test is run to check the chemical constitution and stability of the particular pigment involved. Routine microscopic observation is used to determine uniformity of particle size.

Fineness:

Particle size of pigments must be given particular attention and may be considered under two headings. The first has to do with contamination by coarse particles and can best be referred to as macroscopic fineness, while the other is concerned with the size of the average particle which is determined microscopically. In regard to presence of coarse particles, it is well-known fact that flaws or faults due to grit in rubber goods cause early failure from tear and thereby give low tensile strength and poor tear resistance in laboratory tests. To determine the presence of coarse particles, the amount retained on a 325-mesh screen by the water-wash method is usually measured. For soft rubber, the specification should permit not more than 1 per cent retained on a 325-mesh screen. Much better mixing conditions can be obtained when a coarser pigment is used, but poor flex resistance will result if a pigment with too coarse particles is used in soft rubber. Because of the fact that hard rubber does not undergo flexing, it is possible to take advantage of the easy mixing of a somewhat coarser pigment in compounding for hard-rubber articles. Most hard-rubber batches are very soft in the uncured state because of the softening effect of large amounts of sulfur and the presence of considerable added softener in most cheap batches. This softness will allow very fine pigment to agglomerate during mixing, and pigment lumps will result in the cured stock. By using a slightly coarser pigment with a controlled particle size these mixing conditions are improved. This control is obtained by specifying the maximum and minimum retained on 100, 200 and 325-mesh screens. Requirements similar to those for pigments used in hard rubber apply to sulfur for use in soft rubber. Sulfur dissolves in rubber if only fairly fine and mixes easily into the batches. Hence specifications for sulfur may require not more than 95 per cent and not less than 85 per cent to pass through a 200-mesh screen, but 100 per cent through a 100-mesh screen. As the average particle size diminishes in microscopic fineness, more and more difficulty is encountered in obtaining uniform distribution between a soft binder phase and the fine pigment. Pigments having the smallest average particle size give much trouble both on the mixing mill and in the internal mixer. In this respect all extremely fine pigments are like zinc oxide. A close balance must be maintained between the advantages of fine pigments in producing rubber compounds of superior physical properties and the difficulties encountered in mixing. The ability of the miscellaneous fillers to reinforce rubber compounds is also a function of the particle size, finer pigments giving greater stiffness, higher tensile strength, and more tear resistance.

Effects on Vulcanization and Aging:

Most pigments that are desirable for use as fillers have no effect on rate of vulcanization or aging unless chemical impurities are present. However, lime, litharge and magnesia are inorganic accelerators of vulcanization and, as such, do not fall into the general class of fillers. These materials are often used in rubber goods without organic accelerator or zinc oxide; but when used in conjunction with either of the latter materials they enhance the accelerating effect. It is not the purpose of this paper to discuss the theories of vulcanization, which have been very well treated by Bedford[1] and a number of other investigators in the past fifteen years. It is sufficient here to mention that these pigments, functioning as accelerators, are active only at the surface of the particles and probably after solution in the rubber mix, by small amounts of fatty acid. It is not surprising, therefore, that differences in rate of cure with these pigments may be observed if samples of different particle size are tested. The most striking example of this is the case of magnesium oxide. One material in quite general use is prepared by calcining natural magnesium carbonate. The product, known as heavy calcined magnesia, is finely ground and is an active accelerator in both hard and soft rubber when used in concentrations of about four or five parts by weight on 100 parts of rubber. Another magnesium oxide is prepared by calcining chemically precipitated magnesium carbonate. This oxide is extremely active as an accelerator of vulcanization in concentrations of one-half to one part by weight in 100 parts of rubber. An examination of these two materials under the microscope indicates a wide divergence of particle size: the more active material, known as light calcined magnesia, being the finer of the two. The same differences can be discovered if an examination of the lead oxides of different activity is made or if calcium oxide is examined in the same way. It has, therefore, become standard practice to make a microscopic examination of these pigments as part of the testing routine. In addition to the analyses to which every pigment is subjected, magnesium oxide is always tested carefully for manganese because of the close association of these two metals in nature and the deleterious effect of manganese on aging.[1]

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[1] C. W. Bedford and Winfield Scott, "Vulcanization," *Industrial and Engineering Chemistry*, Vol. 13, No. 2, p. 125.

At the present time, practically all pigments are tested for manganese, a metal which has such wide distribution that minute amounts are found in many naturally occurring minerals. A rubber compound which would ordinarily have a life of several years may be completely destroyed in a year or less due to the presence of manganese in an active form. The natural aging effects are not as well authenticated as the accelerated tests such as oxygen bomb aging and hot-air oven tests, but it has been demonstrated in enough cases that natural aging follows these accelerated tests to know that manganese, like copper and cobalt, is not a welcome impurity in any rubber compounding ingredient. Fortunately, the presence of copper and cobalt is easily controlled since they are present in few of our naturally occurring minerals and must usually be introduced through some process in the manufacture of the finished pigment.

Special Properties:

Some pigments produce special physical properties in rubber. Both diatomaceous earth and magnesium carbonate, because of their particle shape, impart stiffness to rubber compounds. These materials are used only where a special type of hardness or stiffness is desired.[2] To measure these properties, tests of transverse strength, permanent set and stress-strain characteristics are specified. The dry powders most generally used do not affect human beings in the factory or in the finished goods during use. Most of them have no odor or toxicity (except lead compounds) and are usually agreeable to handle.

Materials added to rubber batches for the purpose of producing color form a very special class of compounding ingredients. The major portion of the testing of these materials is done in a particular type of compounded rubber containing either lithopone or titanium oxide to give a background for the color. Only a small percentage of the color is used in this test recipe. After a color has been approved as satisfactory on a tinting strength - cost basis, it is necessary to submit it to a number of special tests to determine whether it is satisfactory for general use in rubber goods. Coloring material should not be soluble in rubber for this results in a tendency for the dye to bleed from the colored portion into any white material touching it. Even though the color is not one which bleeds readily, it still may be soluble enough to bloom on to the surface of a dark-colored portion of rubber either overlying or adjacent to the colored section. This consideration is important in footwear manufacture. The effect of the all-weather aging test, usually run on the roof in direct sunlight, and the effect of sunlight through glass are quite important in determining how long the bright appearance of a piece of colored rubber will last in a particular type of service. Most colors are also tested by soaking the rubber sample in water to see whether the organic dye involved will color the water. Some coloring ingredients in rubber are even tested in ethyl alcohol, ether, or vegetable oils where there is a possibility that these colors will go into druggists' sundries and be used in contact with a variety of medicinal solutions. The evaluation of a color for rubber has been based upon so many tests that it is not difficult to understand why only a very limited number of organic dyes are satisfactory for use in rubber goods. In general the mineral colors are most satisfactory on every point except strength and brightness. Iron oxide, chromium oxide, cadmium sulfide and ultramarine blue are fast to light and to many chemicals and may be safely used for most types of service. The amount of red oxide of iron used by the rubber industry, however, exceeds the total tonnage of all other colors. It must be very carefully tested for manganese in order to be certain that it will not produce poor aging.

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[1] B. S. Taylor and W. N. Jones, "Effect of Certain Metallic Salts on the Aging of a Tread Compound," *Industrial and Engineering Chemistry*, Vol. 20, No. 2, February, 1928, p. 132.

[2] R. H. Seeds, "Compounding: A Discussion of the Fillers, Reinforcing Agents and Softeners in the Compounding of Rubber," Symposium on Rubber, Cleveland Regional Meeting, Am. Testing Mats., p. 46 (1932).

Pigments Other Than Dry Powders:

Liquid or resinous compounding ingredients, known as softeners, are used for the prime purpose of improving the processing of uncured rubber stocks. Specifications written for these materials very seldom involve any standard tests in vulcanized rubber because their effect on cured rubber is often quite different from the properties which they impart in the unvulcanized mix. Each softener has its own purpose in rubber, and rubber compounders can describe the function of the softener used in any given compound, but it has been found difficult to create a rubber test which could be used as a control for these materials. In the inspection and testing of softeners, an attempt is made to control any material used in rubber according to empirical rules. The heating loss at 105 C. (221 F.) and the percentage of ash are general tests to determine the uniformity of the material. In applying the five critical points to the testing of softeners, the solid softeners, such as mineral rubber or hardwood pitch, are easy to incorporate but as more liquid softeners are chosen, difficulties of incorporation occur. This seems to be primarily a function of the greasing action of the softener on the mill roll during the mixing process. Mineral rubber and hardwood pitch both go into rubber easily and make the unvulcanized rubber compound mushy so that it can be run through an extruding machine or be formed readily. However, neither of these materials soften the cured rubber to any great degree. Paraffin, petrolatum and mineral oil aid in processing of uncured stock, and in addition soften the cured rubber. The fatty acids and vegetable oils have a somewhat different type of softening effect in uncured rubber and vary in the degree of softening imparted to cured rubber.

Each softener has its own effect on the rate of vulcanization and aging. Iodine number as a measure of unsaturation may be used to indicate their possible behavior. A highly unsaturated material such as asphaltum oil absorbs sulfur during vulcanization and thereby retards the rate of cure. Material such as linseed oil, because of the unsaturated residue remaining after vulcanization, has a harmful effect upon aging. Most accelerators are affected by the amount of organic acid present in the rubber mix. Among the most active in speeding vulcanization with some organic accelerators is stearic acid. This property is utilized for the purpose of activating organic accelerators. Therefore, in the testing of fats or oils such as cottonseed oil, palm oil, rapeseed oil, brown grease and other natural fats, a very important test is the determination of acid number. Saponification number is used to indicate whether diluents have been added in the form of mineral oils. A great many softeners are controlled by determining the degree of solubility in organic solvents such as benzol or gasoline—a general test to measure the amount of foreign material. Melting point or softening point is obtained by a number of methods and it is customary to use the test preferred by the supplier or the other major industries which consume the same material.

Considerations of mixing ability, rate of vulcanization, reinforcement quality, special physical properties and personnel factors are the five important criteria upon which to judge pigments for use in rubber goods. Through development of suitable specifications covering properties quite readily measured, it is entirely possible to obtain pigments of uniform quality and thereby avoid many day-to-day fluctuations in the manufacture and performance of rubber goods.

MATERIALS FOR USE IN STRUCTURAL ENGINEERING MODELS, WITH SPECIAL REFERENCE TO RUBBER COMPOUNDS

By A. V. Karpov[1]

Synopsis

In this paper are discussed the present tendencies toward increased use of engineering models in solving structural problems. The specifications for materials for such models are drawn based on the similarity conditions that a model has to satisfy. The materials presently available are discussed as well as the absence of an ideal model material.

Work is described, done in finding a material suitable for an arch dam model that resulted in the developing of the rubber litharge compound used in the models of Calderwood and Boulder dams. The advisability of developing new materials and the possibilities of rubber compounds are discussed.

Introduction

The maximum stress occurring under service conditions at any point of an engineering structure is one of the main criteria in determining the safety and adequacy of the design. Consequently the determination of the stress at each point of a structure is the most important problem of structural engineering. If the stress can be determined then the structure not only can be designed safely, but can be so shaped that the material is properly utilized, resulting in an economical design.

In structural engineering there are no exact methods by which the stress of a structure can be estimated. Each method is approximate and one of the important problems is to determine how closely every method used or proposed approaches the actual conditions. In the past a most essential factor in a design method was simplicity, which rendered quick application possible by engineers having a limited amount of theoretical knowledge. In order to accomplish this end, assumptions were introduced to simplify the theoretical solutions. So long as the structure or its elements are such that no abrupt change in conditions occurs, the stress evaluated by use of simplified methods may approach closely the actual stress. Abrupt change in conditions, as for instance at the points of application of concentrated loads or at points of change of the cross-section, usually results in high stress that cannot be determined by the use of the conventional simplified methods. In the past, large divergency between design and actual stress was considered satisfactory, the safety of the structure being assured by using large safety factors. With the increase in magnitude of structures came the realization that even an excessive safety factor does not guarantee the safety of the structure if the design methods are crude. To prevent the failure of a structure, it is not sufficient to provide for a sufficiently large average safety factor but it is necessary to have as well a reasonable safety factor at the weakest point of the structure.

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[1] Designing Engineer, Aluminum Company of America, Pittsburgh, Pa.

The most modern aeronautic designs in which the saving of weight is of utmost importance brought out the fact that by providing well-balanced designs even with a comparatively small safety factor a structure may be created which is safe and economical, so far as weight is concerned. This means that there are no elements in the structure in which the safety factor will diverge considerably from the accepted average.

To make a modern, safe, well-balanced and economical design, it is necessary to be able to evaluate the stress and strain with a degree of accuracy much higher than was considered necessary in the past. This requirement of high accuracy in design, which was first realized in connection with aeronautic designs, is at present becoming of importance in every field of structural engineering.

The simplest method of ascertaining how close the actual conditions are to the design is, of course, to test the structure under service conditions after it is built. Practically such tests can in most cases be used only to obtain final confirmations of design method. If such tests were to disclose a considerable divergency in conditions it would be very seldom that changes could be made and the conditions improved. The present method is to make studies on models that can be built, tested, changed and improved with much smaller expenditure of time and money.

Models.—A model is a representation of a prototype which differs from it at least in one, but more often in many, respects. The most common difference is in size, the model usually being a small-scale representation of the prototype.

The testing of a model in general will disclose a behavior that is not identical with the behavior of the prototype. There is a certain relationship between the behavior of the model and that of its prototype which forms a part of a set of similarity conditions. The similarity conditions cover the whole range of the model-prototype relationship and may become very complicated. In designing and building a model, the main problem is to obtain the simplest possible set of similarity conditions consistent with the purpose and practicability of the model, so that from measurements made on the model the exact behavior of the prototype, under different service conditions, may be determined. A study of the similarity conditions will disclose that it is impossible to obtain a theoretically correct engineering model by changing the prototype in one respect only. Simply building the model as a small-scale representation of the prototype, using the same material or materials, will not give a theoretically exact engineering model. If one condition, like the scale, is changed, some other compensating change is necessary to retain the proper model-prototype relationship in behavior.

In structural models generally a change in scale may be compensated by a change of the physical properties of the material. As discussed elsewhere,[1] a model should be built of a material, the modulus of elasticity of which has definite relationship to the modulus of elasticity of the prototype. The most common case is that in which the ratio of the modulus of elasticity of the materials of the model and prototype at corresponding stresses is equal to the scale of the model. Since practically all structural models are small-scale models, this means that the material of the model should have a lower modulus of elasticity than that of the prototype. Every model must not only satisfy the theoretical requirements but must also be practical in permitting the taking of reliable measurements with reasonable ease. Low modulus of elasticity of the material of the model is probably one of the most important factors in making a model practical. Both theoretical and practical considerations favor the use of model materials of low modulus of elasticity. In many cases the practical considerations may be of such importance as to force the use of a material having a much lower modulus of elasticity than the theoretical consideration would require.

IDEAL MATERIAL FOR MODELS OR ARCH DAMS

It is very seldom that a model can be built that will satisfy every theoretical requirement. In most cases an actual model is a reasonable approach to the theoretical, fulfilling a larger or smaller number of requirements depending on the purpose of the model. In this uncertainity lies the danger of improper application of models. By disregarding unimportant factors an entirely satisfactory model may be built even if it does not fulfill all similarity requirements. By disregarding even a single important requirement the model may become unsatisfactory even though it fulfills a number of the other requirements.

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[1] Discussion by A. V. Karpov of paper by B. F. Groat, "Theory of Similarity and Models," *Transactions*, Am. Soc. Civil Engrs., Vol. 96, p. 315 (1932).

The models of dams and particularly of arch dams are probably the most exacting so far as concerns the requirements that must be satisfied by a model material. A material that meets all the requirements in these cases could be characterized as an ideal material for structural models. In order to satisfy the similarity conditions, the model of a dam should be loaded with water and the material of the model should:

1. Have the same specific gravity as the material of the prototype.
2. Exhibit a relationship between stress and strain within the range of stress to which it will be subjected, similar to the stress-strain relationship of the material of the prototype at the corresponding range of stress.
3. Have the same Poisson's ratio as the material of the prototype at the corresponding range of stress.
4. Have elastic properties similar to that of the material of the prototype at the corresponding range of stress.
5. Have a modulus of elasticity considerably lower than the material of the prototype.
6. Have a uniform coefficient of thermal expansion.
7. Have properties that will permit the material to be provided readily in the required shapes.

In general, the material for a small-scale model of a dam could be characterized as a small-scale representation of the material of the prototype. This small-scale representation should have the same specific gravity and display all the properties of the material of the prototype at a range of stress reduced by the scale ratio and a modulus of elasticity reduced by a still larger ratio.

Acceptable Modifications of the Ideal Model Material:

Depending on the purpose of the model, the material may be modified in certain respects. The most significant modification is the specific gravity requirement. In models of bulky structures such as dams or retaining walls in which the weight of the material is an important factor so far as behavior of the structure and its stress are concerned, the specific gravity requirement is of particular importance. In a very large number of structures, such as different types of bridges or trusses, the weight of the material does not play such an important part and with models of such structures, this factor can be introduced if necessary simply by loading the model with additional weights. Under these circumstances the material of the model may have a specific gravity differing from that of the prototype but preferably lower.

Every structural material has a certain degree of non-uniformity. Some of the metals are very uniform while the bulk materials like concrete are much less so. In general, the model material should show a much higher degree of uniformity than the material of the prototype, particularly if strain measurements are to be made. For models of bulky structures the material should be uniform in all three directions though there may be other cases, as bridge or truss models, in which the uniformity is of importance only in one or in two directions.

In a number of models and particularly in models of arch dams, it is of importance to present in the model not only the structure proper, but also the foundation on which it rests. The material used to represent the foundation in the model should be a small-scale representation of the actual ground. Very often the arch dam rests on bed rock, the elastic properties of which are similar to that of the concrete of the dam. In such cases, the similarity conditions will be reasonably satisfied if in building the model the same material is used to represent the foundations and the dam proper.

AVAILABLE MODEL MATERIALS

Arranging the different materials that have been used for small-scale structural models, in accordance with the average values of the modulus of elasticity, the following table will result:

	MODULUS OF ELASTICITY, LB. PER SQ. IN.	WEIGHT, LB. PER CU. FT.	SPECIFIC GRAVITY
Steel	29 000 000	490	7.8
Aluminum alloys	10 000 000	170	2.7
Magnesium alloys	6 500 000	113	1.8
Concrete	from 5 000 000		
	to 3 000 000	150	2.4
Wood	1 200 000	30	0.5
Various synthetic compositions	from 1 000 000		from 0.5
	to 200		to 3.0

Steel:

Steel is sometimes chosen for models of steel structures. The ease in obtaining the material and its low price are obvious advantages. The drawbacks are the high modulus of elasticity, the non-uniformity of the commercially available steel grades and the difficulty of building small-scale models with the very small thicknesses required for the individual members. Professor Beggs overcame the last difficulty by using thin sheets of stainless steel. A very successful model of the Golden Gate Bridge Tower was recently built in Princeton using this material and assembling the model by spot welding.

Metals of Low Modulus of Elasticity:

The aluminum and magnesium alloys are probably the only metals in this class available at present. For those models of steel structures in which the specific gravity is of no concern, these alloys may be very satisfactory. The low modulus of elasticity and the high uniformity of the commercially available alloys are important advantages. However attention must be paid to the strain-stress relationship which for some alloys may be unsatisfactory. A very satisfactory model of a large-diameter steel pipe line was recently built of an aluminum alloy.[1]

Concrete:

Concrete was used as material for arch dam models. The high modulus of elasticity and the non-uniformity of the model material makes it unsuitable for small-scale models. The best known large-scale model is the Stevenson Creek dam which was built by the Engineering Foundation.[2] In small-scale models in order to overcome the drawback of the high modulus of elasticity mercury is usually substituted for water. That increases the deflections but violates grossly the similarity conditions.

Synthetic Compositions:

The above table shows that after excluding wood on account of its non-uniformity and very pronounced directional properties, the choice of available non-synthetic materials is so limited and their moduli of elasticity are so high that it is necessary to make use of synthetic materials. In spite of the large number of such materials, especially the ones developed during the past few decades, the number of compositions that can be used for structural models is very limited. No composition is known that could be used universally, the application of the different compositions being limited to models for the study of particular problems. Practically all of the available compositions fail to meet the specific gravity requirements. In the past few years celluloid, glass and bakelite have grown in importance in connection with photo-elastic stress studies. The drawback of these materials, in addition to insufficient specific gravity, is the non-uniformity which is expressed in building up of internal stress. If such materials are used to build models of metal structures, their stress-strain relationship and pronounced plastic properties are in many cases highly unsatisfactory.

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[1] Discussion by R. L. Templin and R. G. Sturm of paper by Herman Schorer, "Design of Large Pipe Lines," *Transactions*, Am. Soc. Civil Engrs., Vol. 98, p. 154 (1933).

[2] "Arch Dam Investigation," *Proceedings*, Am. Soc. Civil Engrs., May, 1928, Part 3.

Synthetic Compositions for Models of Arch Dams.—Since models of arch dams built of concrete proved unsatisfactory due to the high modulus of elasticity of the material, celluloid models were built. Then the next model (first model of Boulder Dam) was built using a mixture of Acme finishing plaster, cellite (diatomaceous earth) and water. After setting, the resulting material had an average modulus of elasticity of about 100,000 lb. per sq. in. and a specific gravity of about 0.7.[1] The material proved nonuniform. Since this model was loaded with mercury the specific gravity requirement was grossly violated.

The next model was built of a rubber litharge composition developed for that particular purpose. This development was made possible by the cooperation of the Research Laboratory of the Goodyear Tire and Rubber Co. and the Aluminum Research Laboratories,[2] and will be discussed in detail. The material produced was used in a model of Calderwood Arch Dam and in the second model of Boulder Dam. The specific gravity of this material was the same as that of the concrete of the prototype and since the models were loaded with water, one of the most cardinal similarity conditions was satisfied.

RECONNAISSANCE OF MATERIALS SUITABLE FOR MODELS OF CONCRETE DAMS

The material for a model of a concrete dam has to meet the following specifications, which are directly derived from the general specifications for an ideal model material:

1. Specific gravity of 2.4.
2. Straight line stress-strain relationship at the low stress to which the material of the model must be subjected.
3. Poisson's ratio between 0.15 and 0.20.
4. Absence of directional and plastic properties.
5. Low and uniform modulus of elasticity.
6. Uniform coefficient of thermal expansion.
7. Properties permitting the furnishing of the material in the required irregular shapes.

The range of stress to be considered is rather low. For the 1:50 scale model of Calderwood dam, the probable stress was from

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[1] J. L. Savage and Ivan E. Houk, "Model Tests Confirm Design of Hoover Dam," *Engineering News-Record*, April 7, 1934, p. 494.

[2] A. V. Karpov and R. L. Templin, "Model of Calderwood Arch Dam," *Proceedings*, Am. Soc. Civil Engrs., December, 1933, p. 1565.

6 lb. per sq. in. in tension to 20 lb. per sq. in. in compression. For the 1:180 scale model of Boulder dam, the stress range will be correspondingly lower.

On this basis, a preliminary search was made among materials available commercially. No material could be found that would satisfy the specifications entirely. Many materials can be found that will satisfy a single requirement of the specifications, but the number decreases with astounding rapidity with each additional requirement. The combination of the specific gravity of 2.4 and low modulus of elasticity precludes a large number of well-known and commercially available elastic materials, most of them having low moduli of elasticity accompanied by low specific gravities.

Many of the materials developed recently having pyrozylin, cellulose, and cellulose acetate bases, and similar resin bases, have specific gravities much lower than 2.4 and very many of them have pronounced plastic properties. A few attempts were made to load some of these materials to the required specific gravity by using commercially available fillers. These attempts disclosed the fact that many of the materials could not be loaded at all, and in other cases a simple loading of such kind usually resulted in a considerable increase of the plastic properties and the modulus of elasticity. Bakelite, which is so well known commercially, could be successfully loaded to the necessary specific gravity but had to be excluded on account of high skin stresses and the prohibitive cost of manufacturing the different shapes in special molds.

Most of the usual rubber compounds have low specific gravities and rather uncertain elastic properties at low stress but this reconnaissance work showed that, although there was no satisfactory material available, the rubber compounds offered most promise.

RESEARCH IN RUBBER COMPOUNDS

The customary researches concerning elastic properties of rubber are based on tests made in the region of high tension stresses. In the usual commercial investigations, the ultimate tensile stress is the characteristic which receives the most attention. Very little is done in investigating elastic properties in the region of low tensile and particularly compressive stress and no standard apparatus is available for such work. It is necessary to design specimens, testing procedure and special testing apparatus, which have been described elsewhere.[1]

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[1] R. L. Templin and R. G. Sturm, "Methods for Determining the Physical Properties of Certain Rubber Compounds at Low Stresses," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part II, p. 892 (1931).

The specific gravity of 2.4 was considered as a cardinal requirement to be satisfied by all compounds investigated.

In order to exclude, so far as possible, rubber compounds with pronounced directional properties, isotropic fillers or fillers of unknown isotropic properties were used and anisotropic fillers were excluded. Flexibility in compounding was attained by using either one or two fillers so that a considerable variation of the amount of fillers was possible in obtaining compounds of desired specific gravity. The next important point to be decided upon was the shape in which the rubber compound was to be supplied for the model. It was realized that it was impossible, so far as cost is concerned, to fabricate the material in blocks of the many irregular shapes that were necessary for building the model and that it would be necessary to manufacture the material in some uniform shape and bring it afterwards to the required irregular shapes.

Two methods consistent with the present-day manufacturing procedure for rubber compounds were possible: The material could be delivered in sheets or slabs of moderate thickness or in blocks of larger dimensions. Considering the peculiarities of rubber manufacturing, the considerable uncertainty about the possible internal and skin stresses and the possible non-uniform elastic properties to be expected in larger size rubber blocks, it was decided to have the material delivered in slabs of uniform thickness. This thickness was arbitrarily set as 1 in., which is a convenient thickness from the manufacturing point of view. The possible slight variations of the thicknesses of such slabs are not likely to have a pronounced influence on the elastic properties of the material.

Samples for testing were supplied in the shape of slabs, cylinders, or disks. The slabs were of the same 1-in. thickness as the material to be delivered finally, and in order to prepare sample specimens to be used for determining the physical properties, they had to be cemented together into stacks of the necessary height and machined. The fabrication of such slabs and the preparation of test specimens required considerable time, and in order to expedite the work preliminary investigations were made on compounds in the shape of cylinders about 2 in. in diameter and about 6 in. long, which could be fabricated and machined with minimum expenditure of time and money.

Directional Properties:

After a number of compounds were produced, with relatively constant values of the modulus of elasticity and Poisson's ratio, additional tests were run to disclose the elastic properties in different directions. This was done by repeating the samples, fabricating them in sheets and machining three test cylinders for each specimen.

The first test cylinder was made in the same way as the original ones, by cementing together a number of sheets and cutting out a cylinder with its axis at right angles to the surface of the sheets. Such cylinders were designated as "across the slabs" and are indicated on the tables and figures as "X."

The second test cylinder was made by cutting it out of a number of sheets cemented together, with the axis of the cylinder parallel to the surface of the sheets. In some of the samples that were supplied at the later phases of the investigation, the milling direction was indicated on the samples, in which cases the axis of the cylinder was kept parallel to the direction in which the uncured sheets were milled. These cylinders were designated "with slabs 1" and are indicated as "W1."

The third test cylinder was made by cutting it out of a number of sheets cemented together with the axis of the cylinder parallel to the surface of the sheets and at right angles to the direction in which the uncured sheets were milled. These cylinders were designated "with slabs 2" and are indicated as "W2."

When the test of directional properties disclosed that not only the majority of samples showed very pronounced directional properties but also that some samples of the same composition and manufacturing procedure showed very different physical properties, a new phase had to be included in the investigation concerning the possibility of satisfactory repetition of the elastic properties of similar samples. An important fact was brought out which probably is in agreement with the general experience of the rubber industry, namely, that in dealing with rubber compounds at high stresses, the compounds of the same composition and the usual manufacturing procedure will show fairly similar elastic properties while in dealing with rubber compounds at low stresses, compounds of the same composition and very similar manufacturing procedure may show very different elastic properties. The fact that some of the specimens were made in sheets and some in cylinders gave results that could not be compared in spite of the composition, milling and curing procedures being the same.

An attempt was made to eliminate the uncertainties of the curing process by introducing special molds with a piston attachment and manufacturing the specimens in the shape of disks about 6 in. in diameter and about 1 in. thick. In preparing the test cylinders, these disks were treated in the same way as the slabs.

TABLE I.—TESTS OF DIFFERENT COMPOUNDS.

NOTE.—Specific gravity of all compounds = 2.40 ± 0.08.

Specimen (Series R 924 V)	Time of Curing, min.	Temperature of Curing, deg. Fahr.	Shape	Filler by Volume per 100 Volumes of Rubber										Internal or Surface Stresses Noted	Permanent Set, percentage of maximum deformation	Poisson's Ratio, μ	Modulus of Elasticity, E, lb. per sq. in.	Remarks
				Activated Alumina	Barytes	Calcium Aluminate	Carbon Black (P. 33)	Ground Silica	Litharge	Microcell	Thermatonic	Zinc Oxide	Zinc Oxide (American)					
No. 1	75	285	Slab	....	....	....	....	....	22.0	....	....	....	....	None	1.4	0.47	530	
No. 1	75	285	Slab	....	....	....	....	....	22.0	....	....	....	....	None	1.4	0.46	490	Tension test
No. 2	75	285	Cylinder	....	71.5	....	....	....	....	....	....	4.6	....	None	5.4	0.47	1 050	
No. 3	50	285	Slab	....	....	....	....	....	....	....	....	48.0	....	None	5.5	0.33	1 020	
No. 3	50	285	Slab	....	....	....	....	....	....	....	....	48.0	....	None	5.5	0.29	884	Tension test
No. 3c	75	285	Cylinder	....	....	....	....	....	....	....	....	48.0	....	None	6.4	0.49	700	
No. 3d	75	285	Cylinder	....	....	....	....	....	....	....	....	48.0	....	None	8.4	0.51	1 570	
No. 3e	150	285	Cylinder	....	....	....	....	....	....	....	....	48.0	....	None	7.5	0.47	1 050	
No. 5	150	285	Cylinder	....	89.0	....	53.0	....	....	....	....	4.6	....	Slight	15.5	0.34	6 710	
No. 12	100	260	Cylinder	56.0	....	....	....	....	....	....	....	38.7	....	Some	23.0	0.29	2 370	
No. 13	60	285	Cylinder	34.6	59.0	....	....	....	....	....	....	4.6	....	None	17.0	0.48	1 130	
No. 14	60	285	Cylinder	....	67.2	....	....	36.0	....	....	....	4.6	....	None	7.5	0.51	1 500	
No. 15	100	285	Cylinder	61.2	....	....	....	....	13.1	....	....	4.6	....	Very much	24.6	0.40	1 610	
No. 18	60	285	Cylinder	....	....	....	....	....	20.5	40.0	....	4.6	....	Slight	9.0	0.44	1 590	
No. 20	75	285	Cylinder	....	....	....	32.0	....	....	....	....	56.0	....	Very slight	11.0	0.34	7 610	
No. 21	75	285	Cylinder	....	....	....	....	....	....	....	32.0	56.0	....	Slight	6.9	0.43	2 810	
No. 22	75	285	Cylinder	....	....	....	....	....	....	22.0	....	51.0	....	Slight	23.4	0.26	7 410	Time load
No. 22a	150	285	Cylinder	....	....	....	....	....	....	22.0	....	51.0	....	Very slight	9.5	0.48	4 330	
No. 22b	75	285	Cylinder	....	....	....	....	....	....	22.0	....	51.0	....	Slight	13.1	0.43	10 400	
No. 23	75	285	Cylinder	....	....	....	....	....	....	....	....	....	48.0	None	4.2	0.51	1 240	
No. 25	75	285	Cylinder	....	....	30.0	....	....	....	....	....	46.5	....	None	11.6	0.48	5 040	Time load
No. 27	75	285	Cylinder	32.8	....	....	....	....	....	....	....	....	48.0	None	11.2	0.49	800	
No. 28	150	285	Cylinder	....	....	....	....	....	....	....	....	48.0	....	None	8.3	0.50	1 370	
No. 28a	75	285	Cylinder	....	....	....	....	....	....	....	....	48.0	....	None	11.2	0.50	756	
No. 30	75	285	Cylinder	....	....	....	....	....	....	41.0	....	48.0	....	None	27.5	0.42	7 700	

TABLE II.—TESTS TO DETERMINE DIRECTIONAL PROPERTIES.

NOTE.—Specific gravity of all compounds = 2.40 ± 0.08

Specimen (Series R 924 V)	Time of Curing, min.	Temperature of Curing, deg. Fahr.	Shape	Filler by Volume per 100 Volumes of Rubber			Internal or Surface Stresses Noted	Permanent Set, percentage of maximum deformation	Poisson's Ratio, μ			Modulus of Elasticity, E, lb. per sq. in.			Average Modulus of Elasticity, lb. per sq. in.	Deviation of Modulus of Elasticity from Average, per cent		
				Litharge	Microcell	Zinc Oxide			W 1	W 2	X	W 1	W 2	X		W 1	W 2	X
No. 1a	75	285	Slab	22.0	....	....	None	0.5	0.50	0.46	0.48	580	550	475	535	+8.4	+2.8	−11.2
No. 3a	75	285	Slab	....	....	48.0	None	7.0	0.49	0.52	0.19	1 480	2 110	1 660	1 750	−15.4	+20.6	−5.2
No. 3b	75	285	Slab	....	....	48.0	None	8.3	0.48	0.51	0.47	1 550	1 110	950	1 200	+29.2	−7.5	−20.8
No. 30b	75	285	Slab	....	41.0	48.0	Slight	22.0	0.49	0.27	0.17	16 200	29 000	10 200	18 470	−12.3	+57.0	−44.5
No. 31	75	285	Disk	....	41.0	48.0	Some	23.5	0.33	0.38	0.14	7 170	7 550	4 100	6 270	+14.4	+20.4	−34.6
No. 32	75	285	Disk	....	41.0	48.0	Slight	21.1	0.42	0.33	0.54	4 570	6 220	3 170	4 650	−2.6	+33.8	−31.8
No. 33	75	285	Disk	....	41.0	48.0	Slight	14.7	0.42	0.42	0.40	3 510	3 510	970	2 660	+32.0	+32.0	−63.5
No. 34	75	285	Disk	....	41.0	48.0	Slight	23.1	0.40	0.11	0.20	5 100	10 500	4 860	6 820	−25.2	+54.0	−28.8
No. 35	75	285	Disk	....	41.0	48.0	Slight	22.2	0.51	0.32	0.24	2 900	6 140	3 930	4 320	−32.9	+42.2	−9.1
No. 36	75	285	Disk	....	41.0	48.0	Slight	16.2	0.45	0.47	0.37	2 390	2 630	2 360	2 460	−2.8	+6.9	−4.1
No. 41	75	285	Disk	....	36.0	48.0	Very much	14.8	0.39	0.49	[a]	7 130	8 500	[a]	7 820	−8.8	+8.8	[a]
No. 42	75	285	Slab	....	36.0	48.0	Slight	18.8	0.46	0.46	0.44	6 140	9 210	6 110	7 150	−14.2	+28.8	−14.5

[a] Sample badly warped. No test made.

TABLE III.—REPEAT TESTS.

NOTE.—Specific gravity of all compounds = 2.40 ± 0.08.

Specimen (Series R 924 V)	Time of Curing, min.	Temperature of Curing, deg. Fahr.	Shape	Filler by Volume per 100 Volumes of Rubber					Internal or Surface Stresses Noted	Permanent Set, percentage of maximum deformation	Poisson's Ratio, μ			Modulus of Elasticity, E, lb. per sq. in.			Average Modulus of Elasticity, lb. per sq. in.	Deviation of Modulus of Elasticity from Average, per cent			Remarks
				Activated Alumina	Litharge	Microcell	Thermatonic	Zinc Oxide			W 1	W 2	X	W 1	W 2	X		W 1	W 2	X	
No. 1.......	75	285	Slab	....	22.0	....	....	....	None	1.4	....	....	0.47	....	....	530	....	....	....	....	[a]
No. 1.......	75	285	Slab	....	22.0	....	....	....	None	1.8	....	....	0.51	....	....	505	....	....	....	....	[b]
No. 1a......	75	285	Slab	....	22.0	....	....	....	None	0.5	0.50	0.46	0.48	580	550	475	535	+8.4	+2.8	−11.2	[a]
No. 1a......	75	285	Slab	....	22.0	....	....	....	None	1.2	0.49	0.50	0.51	560	560	480	530	+5.1	+5.1	−10.0	[b]
No. 21......	75	285	Cylinder	....	....	....	32.0	56.0	Slight	6.9	....	....	0.43	....	....	2 810	....	....	....	....	[a]
No. 21......	75	285	Cylinder	....	....	....	32.0	56.0	Slight	12.5	....	....	0.51	....	....	2 550	....	....	....	....	Very slow retest
No. 22......	75	285	Cylinder	....	....	22.0	....	51.0	Slight	23.4	....	....	0,26	....	....	7 410	....	....	....	....	[a]
No. 22......	75	285	Cylinder	....	....	22.0	....	51.0	Slight	9.0	....	....	0.54	....	....	11 220	....	....	....	....	Retest
No. 3b......	75	285	Slab	....	....	....	....	48.0	None	8.3	0.48	0.51	0.47	1 550	1 110	950	1 200	+29.2	−7.5	−20.8	[a]
No. 3b......	75	285	Slab	....	....	....	....	48.0	None	8.0	0.48	0.50	0.53	1 570	1 180	1 070	1 270	+23.6	−7.1	−15.8	[b]
No. 3b......	75	285	Slab	....	....	....	....	48.0	None	6.2	0.46	0.49	0.53	1 680	1 210	1 130	1 340	+25.4	−9.7	−15.7	[c]
No. 30b.....	75	285	Slab	....	....	41.0	....	48.0	Slight	22.0	0.49	0.27	0.17	16 200	29 000	10 200	18 470	−12.3	+57.0	−44.5	[a]
No. 30b.....	75	285	Slab	....	....	41.0	....	48.0	None	16.7	0.42	0.39	....	22 650	14 400	....	18 530	+22.2	−22.2	....	[b]
No. 30b.....	75	285	Slab	....	....	41.0	....	48.0	None	20.9	0.42	0.36	0.26	17 300	15 600	14 550	15 820	+9.4	−1.4	−8.0	[c]
No. 30b.....	75	285	Slab	....	....	41.0	....	48.0	None	19.6	0.42	0.41	0.20	15 090	15 500	20 700	17 100	−11.7	−9.4	+21.0	[d]
No. 34......	75	285	Disk	....	....	41.0	....	48.0	Slight	23.1	0.40	0.11	0.20	5 100	10 500	4 860	6 820	−25.2	+54.0	−28.8	[a]
No. 34......	75	285	Disk	....	....	41.0	....	48.0	Slight	19.0	0.33	0.46	0.32	5 830	10 900	4 990	7 240	−19.5	+50.6	−31.1	[b]
No. 36......	75	285	Disk	....	....	41.0	....	48.0	Slight	16.2	0.45	0.47	0.37	2 390	2 630	2 360	2 460	−2.8	+6.9	−4.1	[a]
No. 36......	75	285	Disk	....	....	41.0	....	48.0	Slight	14.5	....	0.47	0.35	....	2 660	2 280	2 470	....	+7.7	−7.7	Retest
No. 36......	75	285	Disk	....	....	41.0	....	48.0	Slight	14.3	....	0.45	0.46	....	2 740	2 680	2 680	....	+2.4	−2.4	Third test
No. 36......	75	285	Disk	....	....	41.0	....	48.0	Slight	13.5	....	....	0.54	....	....	2 320	....	....	....	....	Fourth test
No. 15......	100	285	Cylinder	61.2	13.1	....	....	4.6	Very much	24.6	....	....	0.40	....	....	1 610	....	....	....	....	[a]
No. 15......	100	285	Cylinder	61.2	13.1	....	....	4.6	Very much	8.1	....	....	....	....	....	1 570	....	....	....	....	Retest
No. 12......	100	260	Cylinder	56.0	....	....	....	38.7	Some	23.0	....	....	0.29	....	....	2 370	....	....	....	....	[a]
No. 12......	100	260	Cylinder	56.0	....	....	....	38.7	Some	22.5	....	....	0.36	....	....	2 130	....	....	....	....	Retest

[a] Original test. See Tables I and II.

[b] After first application of hydrostatic pressure.

[c] After second application of hydrostatic pressure.

[d] After third application of hydrostatic pressure.

This special mold consists of a boiler plate $1\frac{1}{2}$ in. thick in which a circular opening 6 in. in diameter was machined. Two pistons, $\frac{1}{4}$ in. thick fitting in the 6 in. opening with a clearance from 0.003 to 0.004 in. were provided to close the opening at top and bottom. The pistons had rounded corners of $\frac{1}{32}$ in. radius. The mold and pistons were heated. The mold with the bottom piston fitted in was then placed on the lower table of a steam-heated hydraulic press and filled with stock, after which the upper piston was fitted and the pressure applied. With a number of stocks the final pressure could be applied immediately. For some stocks a reduced pressure had to be applied for a certain specified time to avoid an extrusion of the material between the mold and piston and only after a certain length of time could the pressure be raised to its final value.

If enough stock was supplied so that the tables of the press could not rest on the mold, then, contrary to the customary procedure during curing, a definite pressure was maintained depending only upon the pressure developed by the hydraulic press.

Elastic Properties of Rubber Compounds:

The results of the original tests of the investigated rubber compounds are given in Table I, together with the composition of the compounds. The results of the tests to determine directional properties are given in Table II and the results of the repeat tests in Table III.[1] Two kinds of repeat tests were made:

1. To check the original tests; and
2. To determine the influence of high pressures on the elastic properties.

The first kind of retests was made on specimens Nos. 12, 15, 21, 22 and 36, and the second was made on specimens 1, 1*a*, 3*b*, 30*b*, 34. The last-mentioned specimens were subjected to 5000 lb. per sq. in. hydrostatic pressure for a period of from 8 to 10 hr., after which they were again tested in the regular manner. On some of the specimens this treatment was repeated several times. All of the specimens were changed considerably except specimens Nos. 1 and 1*a*. It may be noted that specimen 30*b* tended to become more uniform with successive application of pressure but there still remained a wide discrepancy after the third application.

The stress-strain curves for some of these compounds are shown in Fig. 1. These curves are drawn in the conventional way, but the closing line of each of these curves connects two observed points,

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[1] See also discussion by A. V. Karpov and R. L. Templin of paper by H. Hencky, "The Elastic Behavior of Vulcanized Rubber," *Transactions*, Am. Soc. Mechanical Engrs., APM 55 - 8 (1933).

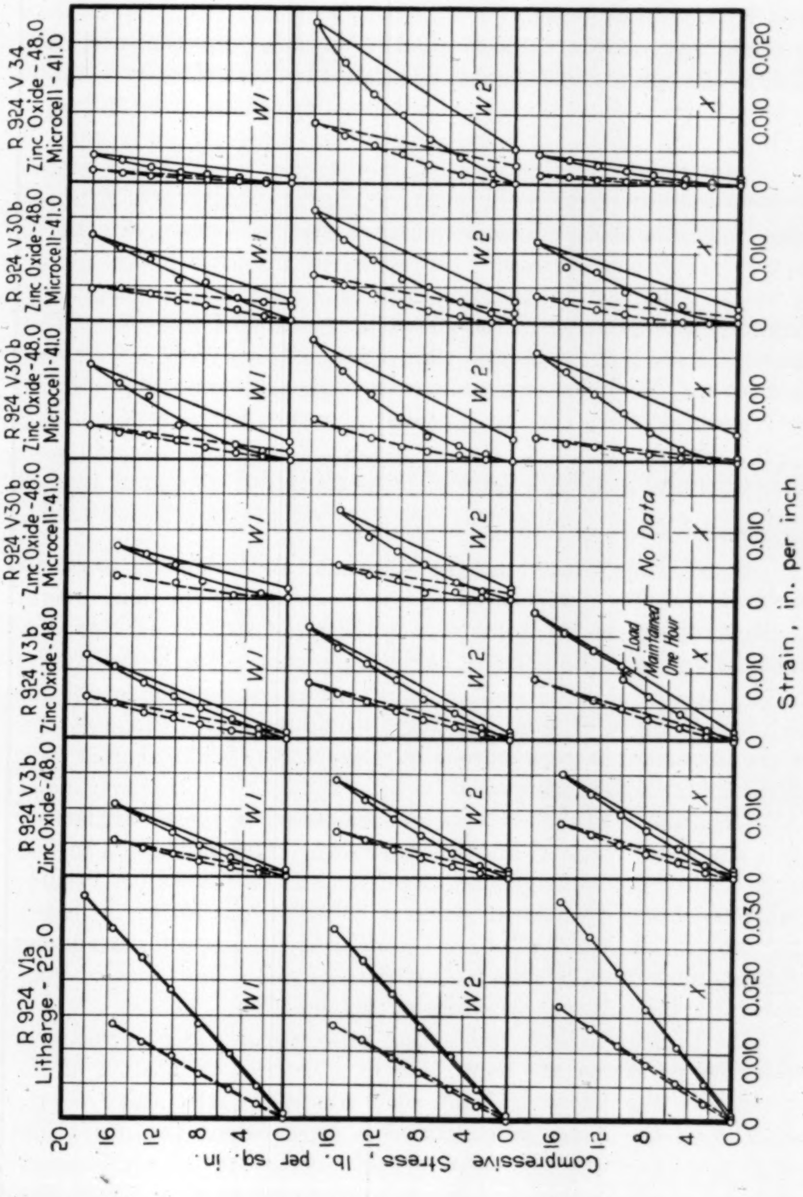

FIG. 1.—Three Directional Stress-Strain Curves for Some of the Compounds.

the largest deformation and the permanent deformation observed after the removal of the loads. These closing lines show the permanent set of the compound.

The solid lines are the strains in the axial directions and the dotted in the lateral directions. The ratios between these two strains at any of the loadings gives the corresponding values of the Poisson's ratio.

From these data it can be seen that the big majority of the investigated compounds display a non-linear stress-strain relationship, a considerable permanent set and very pronounced directional properties. Many compounds, if repeatedly fabricated, show a considerable difference in elastic properties. The most conspicuous exception was the rubber litharge compound designated as V1. This compound has a linear stress-strain relationship, a very low permanent set and a high degree of uniformity so far as directional properties are concerned. It can be repeatedly manufactured without any appreciable change in its elastic properties. The disadvantage of this compound is its high value of Poisson's ratio.

Suggested Research in Rubber Compounds:

Model study concerning the structural and designing problems in which stability is influenced to a large extent by the dead weight of the structure, as for instance in gravity and arch dams, lags very much behind other branches of engineering work. The model studies in this line are not made as often and to the extent that the magnitude and responsibility of such structures would suggest. Probably one of the important factors repressing these studies is the absence of a proper material for such models.

The rubber litharge compound described is more satisfactory than any other material known at present. The problem is to find a material of the same general characteristics as the rubber litharge compound but with a lower value of the Poisson's ratio. It seems that rubber compounds offer most promise in this respect. A number of compounds, unsatisfactory otherwise, showed a low value of Poisson's ratio, indicating possibilities in this direction.

The present milling and curing processes introduce directional properties or exaggerate them, due to a definite orientation of the particles of the fillers. Under such conditions, the shape of the particles of the fillers influences to a large extent the directional properties and even the very limited number of known isotropic fillers will result in more or less non-uniform compounds. Following this line of reasoning, in order to prepare a uniform material the manufacturing procedure should be changed so as to eliminate the undesirable influence of the milling and curing processes. Such changes should not only result in uniform compounds but also increase considerably the number of fillers that could be used. Another problem is the establishing of a more exact manufacturing procedure, so that when the same compound is repeatedly manufactured it will display the same elastic properties.

Based on the work done in developing the rubber litharge compound, the following suggestions can be made:

1. Fillers which during the curing enter in a definite chemical combination with the rubber are preferred. The resulting compounds would be of higher quality as compared with compounds in which fillers are merely mechanical admixtures.

2. Since the Poisson's ratio of rubber is very close to 0.5, a compound of a low Poisson's ratio in all probability will contain fillers in large volumetrical proportions.

3. The main difficulty to be overcome lies in the discovery of a filler or fillers which will permit heavy loading of the compound without impairing its elastic qualities and without introducing pronounced plastic properties.

DISCUSSION

Mr. H. J. Gilkey[1] (*by letter*).—This paper should constitute a challenge to research workers in the field of materials and especially to those who are affiliated with organizations which specialize in developing and marketing new products. The keen and widespread interest shown in the conferences on models recently held under the auspices of the Society for the Promotion of Engineering Education at Cornell University indicates only too clearly the present trend toward increased uses of models as supplementary design aids for important structures. Not only is there a considerable demand for better materials from which the models of various types can be fabricated, but it is probable that materials found suitable for model work will develop other unforeseen adaptations.

The two outstanding model studies of arch dams were started independently by the Aluminum Company of America under Mr. Karpov at Pittsburgh and by the U. S. Bureau of Reclamation in cooperation with the Engineering Foundation at the University of Colorado. In each case the first and most important hurdle to be surmounted was that of selecting a material suitable for the model.

From the standpoint of model testing it can be considered fortunate that the two ventures were entirely independent of one another. It is equally fortunate that in the later stages the Aluminum Company and the Bureau of Reclamation pooled their experiences in the construction and testing of a rubber-litharge model of Boulder Dam.

That neither the engineers of the Aluminum Company nor those of the Bureau of Reclamation were convinced that the material developed by the other was the better[2, 3] is unimportant and rather to be expected. The significant thing is that both materials fall far short of the ideal for a model of an arch dam but that in spite of this fact each of these two outstanding research organizations obtained results which its qualified experts consider to be of value.

An account supplying the background to what has been done may provide something constructively suggestive to one interested in further developing model materials.

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[1] Professor and Head of Department of Theoretical and Applied Mechanics, Iowa State College, Ames, Iowa.

[2] Discussion by A. W. Simonds of paper by A. V. Karpov and R. L. Templin, "Model of Calderwood Arch Dam," *Proceedings*, Am. Soc. Civil Engrs., Part I, April, 1934.

[3] See Karpov's comments on the plaster-celite material on p. 669.

The present paper, supplemented by the other publications cited, supplies in excellent detail the objective set, methods followed and results obtained in developing the rubber-litharge material used by the Aluminum Company. No such detailed narrative has been given for the plaster-celite material developed and used by the Bureau.

The work of the Bureau was performed largely by Fredrik Vogt followed by A. W. Simonds, each of whom functioned under I. E. Houk, Senior Engineer, and J. L. Savage, Chief Designing Engineer. The writer, then located at the University of Colorado, was closely in touch with the work, especially in its earlier stages and must assume the blame for some of the suggestions which led to the evolution of the material finally selected. It is felt that the record should include mention of ventures which promised little and some of which now appear to have been ill-advised, as well as of those which were more successful or appealing, especially if such mention appears to have merit in pointing out what not to do.

In the Bureau's initial model tests, those on the model of Stevenson Creek Dam,[1] the structure was so slender that ample deformations could be obtained by using mercury loading on a concrete model. The weight of the structure itself was a negligible factor and the fact that the weight of the loading liquid used was 13.6 times that of water while the material was of the same weight as that of the prototype did not constitute a serious violation of similarity conditions. Concrete also sufficed to give measurable deformations in the Gibson model[1] although the matter of weight was more important. For the Boulder Canyon model it was apparent that a much more flexible material was required even with the magnified mercury loading. Measurable deformations (under mercury loading), elastic behavior under load and values of Poisson's ratio approximating those for concrete were set as major requirements.

In the preliminary reconnaissance a great many materials were considered, some in detail and others to be dismissed immediately. Among them were: rubber, celluloid, bakelite, maizolith, plastic wood, sulfur (melted and cast in place), leadite, asbestos-concrete, plaster of Paris, concrete in which chunks of rubber were used for coarse aggregate, concrete made porous by using an excess of pea gravel, etc.

Some experiments were made involving tars and asphalts, but the flexibility obtained was plastic rather than elastic. Celluloid was somewhat stiff and had too high a value for Poisson's ratio. Rubber also deforms at approximately constant volume (Poisson's ratio about

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[1] J. L. Savage, I. E. Houk, H. J. Gilkey and Fredrik Vogt, "Arch Dam Investigation, Vol. II," Engineering Foundation, New York City (1934).

0.5) and was considered unsuitable for this and other reasons. Rubber as aggregate in concrete was obviously unsuitable because of the limited deformability of the cement paste and the great deformability of the rubber aggregate. The excess-pea gravel porous concrete also proved unwholesome, being characterized by friability combined with little reduction in stiffness. Initial stresses, hardness and casting or machining difficulties characterized such materials as bakelite and maizolith. So much for the general reconnaissance.

The plaster-celite mixture was evolved as a result of an interesting sequence which will be detailed.

It is well known that the stiffness (modulus of elasticity) of concrete bears a crude relationship to its ultimate strength. The strength bears a definite relationship to the water-cement ratio. It was thought that possibly by increasing the water-cement ratio indefinitely the modulus could be reduced to the very low value desired. Experimentation on this lead resulted, of course, in a very lean mixture which was harsh and friable. To overcome this characteristic, celite was substituted for sand. The resulting mixtures were fat and workable, but the great predominance of very fine material produced cracking from excess shrinkage in curing and drying. At this stage it was recalled that many plasters do not shrink in hardening; in fact, some dental plasters swell. Plaster of Paris, both alone and mixed with portland cement, had previously been tried and found to be too stiff. In the light of these experiences, it was natural and logical to substitute plaster for cement and to experiment further with the celite. Before the final selection was made, extensive experimentation was conducted upon many varieties of plaster and upon the relative proportions of the three ingredients. During the elimination stages, many miniature beams about $\frac{3}{8}$ by 1 in. in cross-section and about a foot long were made and tested in flexure for stiffness, strength and elastic behavior. For the more promising mixtures, larger beams 3 by 3 by 40 in. were made and tested for strength, stiffness, elastic behavior and volume change (as indicated by changes in length). Stress-strain relationships were determined in compression, flexure, torsion and tension for large numbers of specimens, both before and during the construction of the model. In fact, one of the advantages of being able to use a material molded in place was the ease of maintaining close job control by taking as many samples as desired from each pouring.

An unforeseen problem arose, the importance of which proved to be second only to the selection of the material for the model. Plaster of Paris castings behave elastically when thoroughly dried out, but are plastic if free moisture is present. The pouring of the relatively massive Boulder Dam model in stages that made a thorough drying out possible, without resoaking as another layer was added, called for much additional experimentation and ingenuity on the part of the Bureau staff.

The important fact is that a definite need existed and that it was finally met, not by a direct and predetermined technique but rather by a process of running down successive leads and solving a series of problems. At no stage was it expected that all the similarity conditions could be met. For mercury loading the required weight of material would be more than a ton per cubic foot. Obviously no such material was or is in prospect. The plaster-celite mixture at less than one-third the weight of concrete aggravates the difference but even iron would have had less than one-fourth the weight required.

The Bureau found it possible to bring the plaster-celite mixture to the weight of concrete by using bird shot in the mixture. When this was done, there was a surprising uniformity of shot distribution in spite of the very fluid mixture employed. The presence of the shot increased the stiffness somewhat, however, and since even trebling the weight to equal that of concrete still fell far short of the weight needed to meet the conditions of similitude it was decided not to load the material with the shot. In all of the Bureau's model testing, weight has been the condition purposely violated and of which account has had to be taken mathematically or by supplementary experimentation.[1,2]

It was a fortunate coincidence that Poisson's ratio for the plaster-celite material was identical with that of concrete. The material developed and used resembled concrete in several important respects, proved to be satisfactorily uniform, and served its purpose admirably.

Thus, in spite of the unmet need for materials suitable for constructing models of arch dams, two important contemporaneous projects were carried to completion and two widely different materials were developed and used. These two types of model material will doubtless serve as starting points for some of the future investigational work that is sure to be done in an attempt to register further progress. It is hoped, also, that the author's and the writer's descriptions of how the respective materials were evolved may contain something of benefit to others entering the field. Great credit is due Mr. Karpov

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[1] In some recent supplementary tests the Bureau has introduced forces to simulate weight in a very ingenious manner.

[2] E. D. Smith and E. O. Bergman point out that the matter of weight is relatively unimportant, see *Proceedings*, Am. Soc. Civil Engrs., September, 1934, pp. 1025, 1030.

and his co-workers and the staff of the Bureau of Reclamation for the able and ingenious manner in which acceptable solutions were found for the baffling supplementary problems encountered. Each organization is entitled to a justifiable pride in its accomplishments. Nevertheless, there remain great opportunities for improvement by qualified workers whose specialty it is to develop and market materials designed to meet specific needs.

MR. IVAN E. HOUK[1] (*by letter*).—The author has contributed a valuable discussion on the subject of materials for structural engineering models. However, the writer does not agree with some of his statements, particularly as regards similarity requirements, loading methods, and most promising materials for use in models of arch dams.

In the case of engineering models, built for the purpose of studying the magnitude and distribution of stress in prototypes to be constructed with elastic materials, the writer believes that the most important requirement of the model material is that it be uniformly elastic in all directions, so that its action under varying load conditions can be studied by means of mathematical formulas based on the theory of elasticity. It is not sufficient to measure strains in a model and apply a constant multiplier to predict stresses in the prototype, even if the procedure is found to check, or can be made to check, in one or two isolated instances. The investigator must study the fundamental scientific principles involved in the action of both model and prototype, so that he can safely predict the changes in stress magnitude and distribution to be expected as a result of possible changes in design.

The requirement that Poisson's ratio be the same for both model and prototype materials probably ranks next in importance to the elasticity requirement. Effects of small differences in Poisson's ratio can be included as a step in the mathematical analysis of the structure, but large differences may introduce undesirable complications not susceptible of accurate analysis.

As regards model tests of arch dams the writer does not consider the specific gravity factor to be important, either from the standpoint of verifying technical design methods or from the standpoint of making direct predictions of prototype action, unless serious cracking occurs and unless the total cantilever stresses, including both dead and live load effects, are accurately measured in both model and prototype, a procedure which is extremely difficult to follow in the case of the prototype. If only live load effects are being investigated, and cracking does not occur, the specific gravity of the model material is not important and the specific gravity of the loading liquid, if different from unity, can be properly considered in reducing the observations.

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[1] Senior Engineer, U. S. Bureau of Reclamation, Denver, Colo.

The specification of a low modulus of elasticity for the material used in the model construction is important only to the extent that the model strains and deflections be measurable with readily obtainable laboratory equipment. The writer believes that the modulus of elasticity of the litharge-rubber material, developed by the author in connection with his work on Calderwood Dam, is entirely too low to be satisfactory in model tests of arch dams. He believes that serious complicating factors of a detrimental nature are introduced in the tests of litharge-rubber models due to the extreme flexibility of the material and the accompanying volume changes which take place when the loads are applied.

The Bureau of Reclamation, in its model tests of the Stevenson Creek and Gibson dams, conducted at the University of Colorado, Boulder, Colorado, in cooperation with the Engineering Foundation Arch Dam Committee, found concrete models, loaded with mercury, to be entirely satisfactory from the standpoint of investigating design methods.[1] In fact, the tests of the Stevenson Creek model were also satisfactory from the standpoint of direct similarity comparisons, inasmuch as the increased stresses caused by the mercury load produced cracks in the model very similar to those in the prototype. However, in developing plans for model tests of Boulder Dam it was found that owing to the unprecedented size of the prototype a material with a much lower modulus of elasticity than concrete would be necessary, even though the models were to be loaded with mercury and the deformations measured with extremely sensitive optical strain gages. After a lengthy laboratory investigation of all available materials, a plaster-celite mixture was developed which was found to be uniformly elastic in all directions, to have a sufficiently low modulus of elasticity, and to have a Poisson's ratio almost identical with that of concrete. In other words, the material was found to fulfil, in an entirely satisfactory manner, the two most important similarity requirements for model tests of arch dams.

A 1:240 scale model of Boulder Dam was built of the plaster-celite material in the spring of 1931, and was subsequently subjected to more than 1300 mercury loads, to a prolonged mercury load flow test, and to an extremely severe temperature test, the last being made by circulating dry ice vapor along the faces of the model. The model was found to operate satisfactorily throughout the entire program. The results were found to demonstrate the safety and adequacy of the curved-gravity Boulder Dam design, to verify the trial load method of analyzing the action of curved dams, and to supply a wealth of detailed technical information needed in analytical studies of arch dam design. A brief account of the work, including some of the more important results obtained, was published in *Engineering News-Record* about two years ago.[1]

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[1] Engineering Foundation Arch Dam Investigation, Report by Committee, Vol. II, May, 1934, Engineering Foundation, New York City.

After the work on the plaster-celite model was finished it was decided to build a new model, using an entirely different material in order to secure an independent check on the results of the plaster-celite tests. Celluloid and hard rubber were the materials first considered, but both were rejected because of manufacturing difficulties encountered in securing homogeneity in the thicknesses required. It was then decided to use the litharge-rubber compound developed by the Aluminum Company of America. Accordingly a supply of litharge-rubber sheets, 1 in. thick, was purchased from an eastern rubber company, under practically the same specifications used by the Aluminum Company of America in securing materials for their model of Calderwood Dam. A 1:180 scale model of Boulder Dam was built of this material in the winter of 1932 and 1933. This model was subjected to approximately 950 water loads, to a continued water load flow test, and to a severe temperature test, the last being made by cooling and heating the reservoir water. The results of the rubber model investigations checked very closely with the results of the plaster-celite model studies, making proper allowances for differences in physical properties of the two materials, differences in specific gravity of the loading liquids, and differences in depths of supplementary foundations and abutments.

As a result of his experience with the two models of Boulder Dam the writer does not consider the soft rubber material to be nearly as satisfactory a material for use in model tests of arch dams as the plaster-celite material. The rubber, although elastic, is not uniformly elastic in all directions, the moduli of elasticity in the three directions normal to the faces of the test specimens varying as much as 30 per cent due to differences in the grain of the rubber. Consequently the litharge-rubber material does not satisfy the most important similarity requirement. Moreover, the relatively high and variable Poisson's ratio of the rubber, approximately 0.33 to 0.62 in the different directions, averaging about 0.50, violates the next most important similarity requirement. A third disadvantage of the rubber material is its variable coefficient of thermal expansion, different tests showing values ranging from 0.000060 to 0.000090, a total range of 50 per cent. None of these objections can be raised in the case of the plaster-celite material.

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[1] J. L. Savage and I. E. Houk, "Model Tests Confirm Design of Hoover Dam," *Engineering News-Record*, April 7, 1932, pp. 494, 499.

In the writer's opinion the only advantage of the soft rubber material over the plaster-celite material, for use in building models of arch dams, is that in the case of rubber models the loading liquid can be placed directly against the upstream face of the model and strains measured at the upstream face by observations made through the loading liquid. In the case of plaster-celite models water or mercury loads must be applied in a rubber bag, held against the upstream face by a close-fitting bulkhead form. With such a loading arrangement it is hardly feasible to secure satisfactory strain measurements at the upstream face of the model.

The modulus of elasticity is much lower for the litharge-rubber material than for the plaster-celite material. However, it is low enough in the latter case that strains and deflections can easily be measured with readily obtainable laboratory equipment.

One disadvantage of the plaster-celite material is that the model must be built in comparatively thin layers and each layer thoroughly dried before the next is poured. Although this naturally slows up the construction of the model, the total time required for the building operations is not appreciably greater than in the case of a litharge-rubber model of equal dimensions.

Although the plaster-celite material is not ideal, the writer believes it is by far the best material thus far developed for use in model tests of arch dams. He believes that future researches of model materials for use in building models of dams should be conducted with plaster mixtures rather than with rubber compounds.

MR. R. W. CARLSON[1] (*by letter*).—The author has directed attention to an important problem. The designer of a model finds early in his studies that the requirements for exact similitude are so strict that often he must either sacrifice exactness or abandon the model. If he departs too far from exactness, there may be greater uncertainty in the model results than in results obtained analytically. However, he should not strive for exactness in those details which do not affect the behavior of the model.

The density of material in a model may or may not be important. For example, if the model of a dam is to be constructed as an elastic unit with no joints to open and with equal moduli of elasticity in tension and compression, the density enters into the measurements only when dead load stresses are investigated. For live load, the weight of the model exerts substantially the same influence after loading as before and therefore does not affect the measurements. If however, an attempt is made to employ the model in determining dead load stresses, the density must be considered.

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[1] Research Engineer, Engineering Materials Laboratory, University of California, Berkeley, Calif.

The density of the model material becomes most important in the case where a structure cannot be guaranteed to carry tensile stress across joints. In such a case, if the weight of the model bears the correct relation to that of the loading liquid, the model can be employed to demonstrate the effect of opening of joints where tension tends to develop. As a joint opens, the liquid enters and exerts its proportionate pressure only when densities are in correct proportion. The heavy rubber compounds developed by the author offer possibilities for such important studies, especially in connection with dams. The effort to obtain such compounds with more uniform properties and with a lower Poisson's ratio should be continued.

MR. H. A. DEPEW[1] (*by letter*).—Rubber has proved so nearly an ideal material for the construction of model dams that rather than seek new materials I would favor improving the rubber. The first thought was that because of the deviation in Poisson's ratio, acicular zinc oxide would be a desirable compounding ingredient. However, when it was realized that the properties must be the same in all directions, the fallacy of this consideration was evident. It would seem that a microporosity of the rubber would be desirable so that as work is done on the face of the rubber dam, the energy is partly absorbed in compressing the porous rubber, and it would partly be utilized in distorting it.

It might be that suitable microporous products would be obtained by using the general methods that had been developed in Latex compounding.

MR. A. V. KARPOV[2] (*author's closure by letter*).—It is very gratifying that the discussion of the paper brought out so clearly the two different viewpoints with respect to models of arch dams. Mr. Gilkey's discussion gives a well-balanced opinion on the use of the plaster-celite material and the rubber-litharge compound. The history of the development of the plaster-celite material had not been given before and hence is of much interest.

Mr. Houk deserves great credit for the early stages of the use of models in designing arch dams, and in so far as plaster-celite models are concerned his discussion is the first attempt to present this material for free discussions. This should be much appreciated by the profession at large. Nevertheless, it would seem that the available information concerning the use of plaster-celite is still of a very general nature and from his discussion one would conclude that this material is more uniform than the rubber-litharge. However, additional positive data on the subject would be necessary to reach a conclusion as to the superior merit of the plaster-celite model.

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[1] Research Chemist, American Zinc Sales Co., Columbus, Ohio.

[2] Designing Engineer, Aluminum Company of America, Pittsburgh, Pa.

In so far as the rubber-litharge compound is concerned, the accompanying Table I gives comparative data on the material used in the Calderwood and Boulder models. Specimens of the material used in each model were tested in the Aluminum Research Laboratories in New Kensington, Pa., under identical conditions and using the same testing procedure referred to in the paper. The stress-strain curves in each case have the straight line shape so characteristic of this rubber compound and repeated tests showed that after the expiration of one year there was practically no aging effect on the Calderwood material.

TABLE I.—COMPARATIVE TABLE OF PROPERTIES OF THE RUBBER-LITHARGE COMPOUND USED IN BUILDING CALDERWOOD AND BOULDER MODELS.

Specimen	Permanent Set, S, percentage of maximum deformation			Poisson's Ratio, μ			Modulus of Elasticity, E, lb. per sq. in.			Average Permanent Set, S, percentage of maximum deformation	Average Poisson's Ratio, μ	Average Modulus of Elasticity, E, lb. per sq. in.	Deviation of Modulus of Elasticity from Average, per cent		
	W1	W2	X	W1	W2	X	W1	W2	X				W1	W2	X
Machined from Material for Calderwood Model:															
Slab No. 1	0.6	2.3	2.3	0.51	0.50	0.52	565	625	485	1.7	0.50	558	+1.3	+12.0	−13.1
Slab No. 5	1.8	1.6	2.1	0.50	0.53	0.52	640	540	470	1.8	0.52	550	+16.4	−1.8	−14.6
Slab No. 52	0.1	0.6	1.7	0.52	0.52	0.51	630	550	510	0.8	0.52	563	+11.9	−2.3	−9.6
Slab No. 56	1.9	1.2	1.8	0.51	0.50	0.51	630	560	470	1.6	0.51	553	+13.9	+1.3	−15.0
Slab No. 91	1.2	1.7	1.4	0.51	0.51	0.52	670	570	520	1.4	0.51	587	+14.1	−2.9	−11.4
Slab No. 187	0.4	0.8	1.7	0.50	0.52	0.53	660	560	500	1.0	0.52	573	+15.2	−2.3	−12.8
Slab No. 377	1.0	1.3	1.9	0.49	0.46	0.50	645	545	500	1.4	0.48	563	+14.6	−3.2	−11.2
Machined from Material for Boulder Model	2.3	2.6	2.7	0.50	0.53	0.50	455	395	345	2.5	0.51	387	+13.7	−0.5	−13.4

The Boulder model rubber-litharge compound has a lower modulus of elasticity than the compound used in the Calderwood model. This fact was known in the early stages of the manufacture of the Boulder dam model material but it was not then considered objectionable. One of the advantages of rubber compounds is the fact that the modulus of elasticity can be increased or decreased as desired by changing the manufacturing procedure.

In so far as the uniformity of the material is concerned, a ±15 per cent variation of the modulus of elasticity from the average modulus should be considered fairly satisfactory. The accompanying Table I also shows that the values of Poisson's ratio in three directions are practically the same, that is, varying between 0.50 and 0.53 in the Boulder model material. It would be interesting to know if equal or better uniformity can be obtained in the cast plaster-celite material when properly tested. It also would be of considerable interest to know the shape of the stress-strain curve of the plaster-celite material. Unfortunately this important characteristic was not stated.

There is, of course, to be kept in mind, that for a pre-fabricated stable material, like the rubber-litharge compound, it is rather simple to determine its properties by testing a sufficient number of specimens. The conditions are more complicated in the case of a cast material of such elastically unstable character as plaster-celite. As was developed in the discussion, the elastic properties of this material undergo changes, after casting and drying, unless the material is kept continuously dry. Changes in the humidity and temperature of the air probably also affect these properties. Under such conditions it would seem that a reliable method would be to cut a sufficient number of specimens from the model itself, distributing the cuts throughout its entire body and testing them in three directions under different but strictly controlled conditions of humidity and temperature. Any conclusion based upon tests made in only one direction and on small specimens cast separately from the model may be misleading. The comparison of the results of tests made on two kinds of specimens, one kind cast separately from the model during its construction and the other cut from the model, after the testing program is concluded, should give a proper engineering conception of the uniformity of the material and the influence of aging. This aging factor in the plaster-celite material may be of considerable importance.

The author disagrees with Mr. Houk's statement that it is extremely difficult to measure on the prototype the total stress, including both dead and live load effects. It is not only simpler, but much more reliable results are obtained by measuring the total stress in the prototype rather than the live load stress only, as described elsewhere.[1]

The author cannot reconcile the following statements by Mr. Houk: first, that the effect of small differences in Poisson's ratio can be included as a step in the mathematical analysis of the structure but large differences cannot be included; and second, that the results of tests on both models checked very closely after making necessary allowances for the difference in materials and conditions, but the plaster-celite model is superior. These statements apparently are based upon his own intimate knowledge of the subject but, unfortunately, he does not give data to sustain this conclusion. The deformations of the foundations and abutments have a very important bearing on the behavior of an arch dam. In the plaster-celite model these conditions could not be properly represented but were represented in the rubber-litharge model. Therefore, it would seem that in order to have the test results on both models agree, a number of assumptions must be introduced with reference to the plaster-celite model. Considering the ultimate purpose of a model, a fair conclusion would seem to be that both models at least are equally satisfactory, provided the results of tests on both of them are in close agreement.

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[1] A. V. Karpov and R. L. Templin, "Model of Calderwood Arch Dam," *Proceedings*, Am. Soc. Civil Engrs., December, 1933, p. 1598.

The author is in complete agreement with Mr. Houk's statement that "The investigator must study the fundamental scientific principles involved in the action of both model and prototype, so that he can safely predict the changes in stress magnitude and distribution to be expected as a result of possible changes in design,"—which means that a proper engineering model is necessary. There seems to be a radical difference in opinion as to what is an engineering model, and this leads to the different kinds of models advocated by Mr. Houk and by the author. Apparently Mr. Houk considers a model to be satisfactory even if there is no definite and simple relationship between the behavior of the model and prototype. He holds that from such model proof may be obtained regarding some of the design assumptions, and that the behavior of the prototype can be predicted by the application of such theoretical assumptions that apparently are proved by the model test. If this view, which has often been taken in the past, be correct, the design of models would be very much simplified and it would not be necessary to fulfill the majority of the similarity conditions and even the model itself need not necessarily be a scalar representation of the prototype.

On the contrary, the author considers such a point of view to be incompatible with the advanced stress and design ideas. The only reason for building a model is that the general engineering knowledge of the behavior of the actual structure is limited and consequently many of the design assumptions are incorrect. The building of a model whose behavior bears no direct and simple relation to that of the prototype, means the substitution of one more-or-less arbitrary set of assumptions for another, leaving open the question as to which one of these two sets is the more satisfactory. The entire and only *raison d'être* of the similarity conditions is that instead of pyramiding one set of assumptions upon another, they make possible the building of a model whose behavior will have a natural relation to that of the prototype without the necessity of making arbitrary assumptions.

In every branch of modern engineering, successful progressive development is possible only if the criterion be imposed that definite and accurate knowledge be used. A statement is made in the discussion that if only live load effects are investigated and cracking does not occur, the specific gravity of the model material is not important. Although not expressed directly, that statement apparently is based on some premise, such as the assumption that the increment of stress after the application of live load is due only to live load. Even under the present limited knowledge, any such premise should be challenged because after the live load is applied, the increment of stress is due at least to three factors: first, the applied live load; second, the redistribution of the dead load stress; and third, the influence of the foundations, abutments and joints. Since no indication is available as to how each of these factors influences the total results, no proper conclusions can be drawn from a model which has improper specific gravity ratio. This is also the only point on which the author disagrees with Mr. Carlson's discussion, all other points of which are well taken.

To Mr. Depew's discussion the author would add that zinc oxide seems to offer good promise and it is possible that the problem may be solved by adding some ingredient in addition to zinc oxide. There is some reluctance in using porous materials for models where strain measurements are to be made between closely spaced gage points.

In conclusion, in order to make plain the difference between Poisson's ratio *versus* the specific gravity requirement, the author desires to make the following statement:

The mercury loaded plaster-celite model has only 2 per cent of the required weight, neglects the representation of foundations, abutments and joints, and behaves radically different from the prototype; the prediction of the behavior of the prototype can be made only by way of evaluating it on the basis of arbitrary assumptions. No amount of improvement of the material can make a mercury-loaded plaster-celite model meet the requirements of an engineering model.

The water loaded rubber-litharge model, whose only important deficiency is in Poisson's ratio, behaves substantially as the prototype, the difference in behavior being of the order of secondary corrections. Further improvement in the material should make the behavior of this type of model such as to eliminate the necessity even of secondary corrections and make it a true engineering model.

SUBGRADE SOIL TESTING METHODS

By C. A. Hogentogler[1] and E. A. Willis[1]

Synopsis

The degree to which a given soil material exhibits the basic properties of stability, compressibility, elasticity, permeability, and capillarity varies with changes in moisture content, compaction and amount of disturbance from its natural structure. This necessitates the selection of a testing procedure designed to measure the controlling physical properties throughout the range of conditions which will obtain and for the particular part which the soil is to play in the engineering structure.

A great variety of tests may be utilized in the different phases of highway construction. Generally, however, the soils are disturbed and are close enough to the surface to be influenced by climatic changes. They are tested in a dried and powdered state for shrinkage, plasticity, etc., to disclose the characteristics of the soil constituents exclusive of soil structure.

Arbitrary procedures are used to determine the moisture contents at significant states of consistency. These moisture contents, or constants, are directly related to density, volume change, shear resistance and supporting power. The range in constants and the interrelationships between the constants furnish a means of measuring the physical properties of soils and for disclosing characterizing soil constituents such as sand, silt, clay, colloids, mica, diatoms, peat, the flocculating limes and the deflocculating sodas which may be present in a mixture. Surveys, investigations and experience have shown these constituents to have a marked influence upon the construction and maintenance of road surfaces.

Based upon constituent materials, physical properties and performance, uniform subgrades are tentatively classified into eight groups, identified by definite test limits and requiring different design features in roads laid thereon.

Introduction

The U. S. Bureau of Public Roads has investigated extensively the influence of soils on highway construction. Among the important contributors are such organizations as the U. S. Bureau of Chemistry and Soils, Massachusetts Institute of Technology, Ohio State University, University of South Carolina, Iowa State College; the State Highway departments of Oregon, Maryland, Virginia, Michigan, Pennsylvania, Ohio, New Hampshire, Missouri, Minnesota, Iowa, Kansas and Texas; and the following individuals: Henry Shirley, C. M. Upham, F. H. Eno, R. W. Crum, H. F. Clemmer, A. C. Rose,

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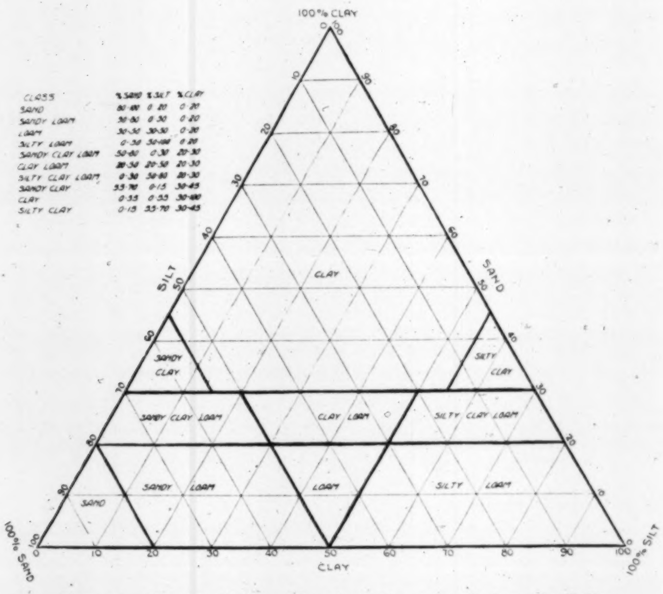
[1] Senior Highway Engineer, and Assistant Highway Engineer, respectively, U. S. Bureau of Public Roads, Washington, D. C.

I. B. Mullis, Charles Terzaghi, Stephen Taber, Victor Burton, A. C. Benkelman, W. I. Watkins, Arthur Casagrande, and Glennon Gilboy.

Reports describing the progress of the work are referred to by boldface numbers in parentheses and listed in the appended list of references.[1] They indicate that the location, the design, and other essential features of highway construction, are in some measure dependent upon the character of the subgrade soil. The practicability of subgrade studies is indicated by a survey made in 1932 by Division 1 of the Committee on Materials of the American Association of State Highway Officials. This survey indicated that 26 states rely in varying degrees on subgrade properties as determining factors in the design of roads and pavements, 13 of which have laboratories equipped to perform all routine tests suggested by the U. S. Bureau of Public Roads. The number with well-equipped soil laboratories has doubled since 1932.



CLASS	% SAND	% SILT	% CLAY
SAND	80-100	0-20	0-20
SANDY LOAM	50-80	0-50	0-20
LOAM	30-50	30-50	0-20
SILTY LOAM	0-50	50-100	0-20
SANDY CLAY LOAM	50-80	0-30	20-30
CLAY LOAM	20-50	20-50	20-30
SILTY CLAY LOAM	0-30	50-80	20-30
SANDY CLAY	55-70	0-15	30-45
CLAY	0-55	0-55	30-100
SILTY CLAY	0-15	55-70	30-45

FIG. 1.—Textural Classification Chart for Subgrade Soils.

THE SOIL PROFILE

Soil occurs in the earth's crust as a series of differently weathered layers known as the "soil profile." Soils of a profile, graded by mechanical analysis are designated as sands, clays, loams, silt loams, etc. Sandy materials are described as "light-textured" and clays as "heavy textured" soils. Figure 1, developed by A. C. Rose of the U. S. Bureau of Public Roads, shows the mechanical grading of the various designations of texture.

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[1] See p. 725.

Profiles developed under the same climatological environment belong to the same soil series. The different series are designated by such names as Chesterfield, Putnam, Berks, Susquehanna, etc.

In well-drained soils of the humid regions the profile consists of a light-textured upper layer designated by the agronomist as the "A" horizon underlaid by a heavier-textured layer known as the "B" horizon. A still lower third layer of unweathered or incompletely weathered geological formation is known as the "C" horizon or parent material. Each horizon or layer may be subdivided, as A_1, A_2, B_1, B_2, etc. For engineering purposes, the various layers may be numbered consecutively from the top down, as shown in Fig. 2, without reference to horizon **(1)**.[1]

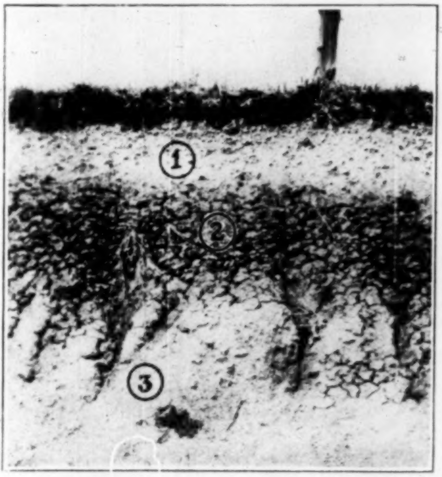

FIG. 2.—Profile of Iredell Silt Loam Showing (1) Lighter-Textured Upper Soil Layer, (2) Heavy, Plastic, Sticky, Waxy Clay, and (3) Decomposed Rock.

Properties of the Same Soil Material May Vary Widely:

During rains and thaws, moisture enters the soil by gravity and forms the ground water which feeds wells and springs. Surface tension, independent of gravity, maintains a moistened layer, termed the "capillary fringe" **(2)**, above the water table. During dry weather, moisture escapes from the surface of the ground by evaporation. Soils swell when moisture enters and shrink when it escapes. They heave during frost and lose stability in subsequent thaws(3,4). Thus to a depth affected by weather conditions, soils are continually changing in properties related to moisture content.

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[1] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 725.

Then, too, the same soil material changes character when disturbed in place, whether above or below the line of seasonal variation; and when consolidated or otherwise manipulated after removal from its place in the soil profile. Eno found change in moisture content up to 124 per cent of the weight of solids due to climatic changes(5). Casagrande reduced the bearing value to as little as one-fifth by remolding a sample of natural clay without changing the moisture content(6). Terzaghi decreased the permeability to 1/75,000 by disturbing the natural structure of soil from Spanish Honduras(7). Saturated samples resisted slaking indefinitely, at the laboratory of the Bureau of Public Roads, while dry samples of the same soils disintegrated quickly when immersed in water(8).

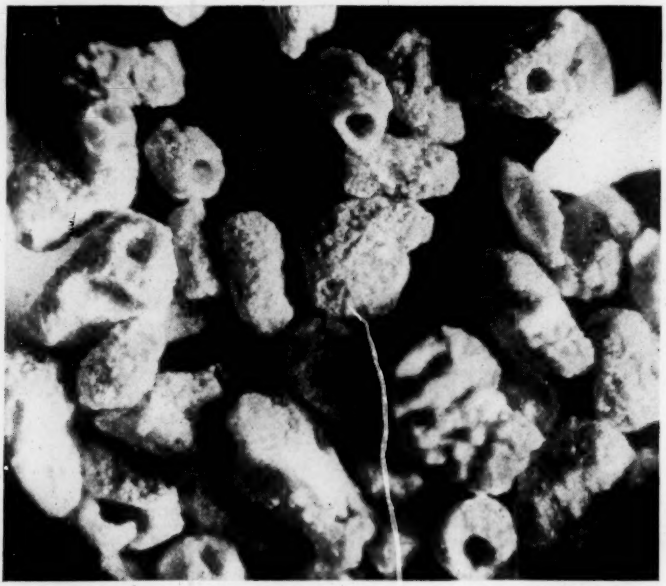


FIG. 3.—Photomicrograph of Loess Soil Particles (× 20).

Loess illustrates the effect of disturbance excellently. The natural structure resists erosion sufficiently to remain almost vertical in cut slopes indefinitely. As subgrade, however, this soil loses stability readily by manipulation in the presence of moisture. According to Pirsson and Schuchert(9), slender vertical tubes, shown broken in Fig. 3, account for the high vertical cleavage. They result from the decay of stems and roots and grasses, growing and buried when successive deposits were forming from dust clouds carried by the wind.

Thus soil does not have well-defined and fairly constant properties like other construction materials, such as concrete, steel, brick and wood. Also, foundation soils react differently from other structures to load. Bridges and buildings fail abruptly when stressed beyond the strength of constituent materials and consequently are designed for safe bearing load. Soils, however, present a different problem. With exceptions, as in the design of earthen dams, the prevention of landslides, etc., abrupt failure is not to be anticipated and resistance to rupture is not an important consideration. Instead, heaving or settlement may damage structures supported by the soil when certain allowable limits are exceeded.

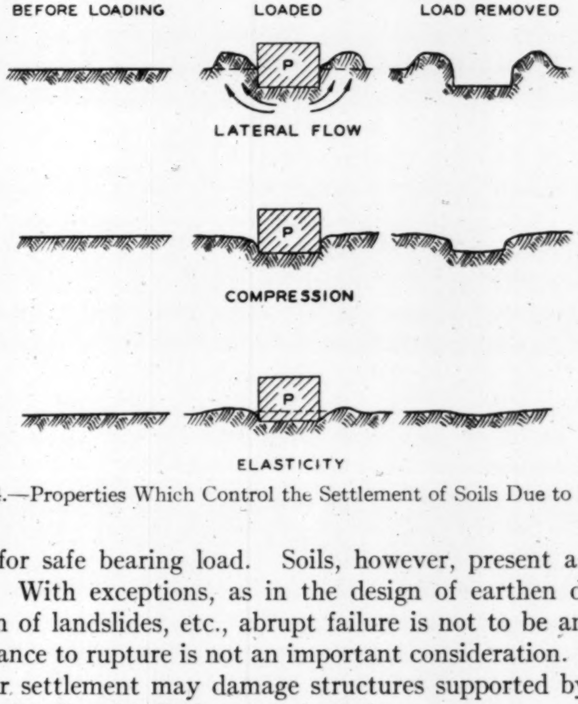


FIG. 4.—Properties Which Control the Settlement of Soils Due to Load.

Therefore footings are designed for the allowable load, designated by the allowable limit of deflection on the stress-strain diagram, instead of the safe bearing load based on the rupturing strength of the soil. Those below the line of seasonal change are designed for deflections produced entirely by load. For foundations above this line, settlement and heave due to causes independent of load are also considered.

BASIC SOIL PROPERTIES

Settlements of soil due to load are caused by the three phenomena—lateral flow, compression, and elasticity—acting individually or in some combination. In lateral flow (see Fig. 4) the soil, without change in moisture content, squeezes out from beneath the load and causes a bulge in the surface adjacent to the load. This deformation remains when the load is removed. The resistance to lateral flow, defined as stability, represents the combined effect of internal friction, or the resistance of soil grains to sliding; and cohesion, the attraction between soil particles(10). Minute moisture films connecting the soil particles, supplemented by possible natural stickiness of colloids, furnish the cohesion.

Compression (Fig. 4) is due entirely to decrease of the porosity of the soil immediately beneath the load. Much of the deformation in this case is also permanent. In elasticity (Fig. 4) the soil displaces laterally under the load in a jelly-like manner without change in porosity, but rebounds upon removal of the load.

The ultimate porosity to which soils compress under load depends upon their capillary properties, and the rate at which they compress depends upon their permeability(8).

Permeability also determines the speed at which the water table rises and falls with alternating wet and dry weather. The height of capillary rise which determines the thickness of the capillary fringe indicates the extent to which soils shrink and swell with changing moisture conditions. The speed of capillary rise determines the rate at which shrinkage and swell occur and the extent to which soils heave due to frost.

Stability, compressibility, elasticity, permeability and capillarity then are the five physical properties(8) which, for engineering purposes, control the performance of soil.

TYPES OF SOIL TESTS

Tests to disclose the physical characteristics responsible for all deformations of soil regardless of causes are of four kinds, as follows:

1. Disturbed soils and those close enough to the surface of the ground to be influenced by change of moisture, temperature, frost, etc., are tested in a dried and powdered state for shrinkage, plasticity and similar properties, which disclose the characteristics of the soil constituents exclusive of soil structure.

2. Soils to be stabilized with admixtures or manipulation in practice are tested in samples prepared according to the construction procedure for properties required in stable fills, subgrades and road surfaces.

3. Soils located below the line of seasonal change are tested by means of undisturbed samples in the laboratory or loading the soil in place in the profile for such engineering properties as compressibility, elasticity and stability which are furnished by both soil structure and soil materials.

4. Soils located below the line of seasonal change, whose structure is disturbed by the penetration of piles or other construction operations, are investigated by loading test piles in place or samples remolded from natural structure and at natural moisture content in the laboratory.

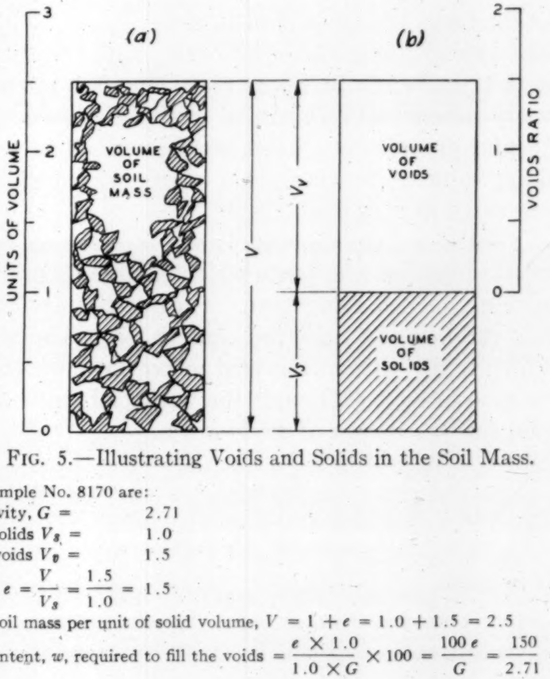


FIG. 5.—Illustrating Voids and Solids in the Soil Mass.

Constants for sample No. 8170 are:

Specific gravity, G = 2.71

Volume of solids V_s = 1.0

Volume of voids V_v = 1.5

Voids ratio, $e = \frac{V}{V_s} = \frac{1.5}{1.0} = 1.5$

Volume of soil mass per unit of solid volume, $V = 1 + e = 1.0 + 1.5 = 2.5$

Moisture content, w, required to fill the voids $= \frac{e \times 1.0}{1.0 \times G} \times 100 = \frac{100\,e}{G} = \frac{150}{2.71} = 55.4$ per cent

Routine Subgrade Tests Disclose Properties of Soil Material:

Tests of all groups may be utilized in the different phases of highway construction. However, only a few have reached the state of development and practical use warranting their publication as tentative standards. They belong to the first group and cover the tests for plasticity, shrinkage and moisture equivalent, which, supplemented by the mechanical analysis, are used in the routine examination of (*a*) subgrade soils; (*b*) soil material in mud jack operations, as binder in sand-clay or gravel roads, or as filler in bituminous road surfacing; (*c*) special surfacing materials such as limerock, caliche and shale; and (*d*) admixtures such as rock powders, diatomaceous earths and bentonite, which may assist in the stabilization of soil road surfaces.

The routine physical tests are not suited for materials containing particles larger than about 0.5 mm. or 0.02 in. in diameter. In practice they are performed upon the fraction passing the No. 40 sieve. The results are referred to as soil constants. The mechanical analysis is made of the fraction passing the No. 10 sieve.

To assist in explaining the significance of the routine tests, the flocculation test(11), the compression test(12), the flow curve(13) and the specific gravity determination are also briefly discussed. Additional tests of the first group which have been described in other reports are for slaking value(14), bearing value of unconfined samples(14), dye adsorption(14) and capillary rise.

In the routine testing procedure, soil is assumed to consist of solids of constant volume and pores which enlarge or shrink when the mass changes volume (see Fig. 5 (*a*)). The density is indicated by the terms "voids ratio" and "moisture content"(8). The voids ratio, e (see Fig. 5 (*b*)), is the ratio of the void or pore volume to that of the solids. The moisture content, w, is the weight of moisture expressed as a percentage of the weight of dried solids.

In completely saturated soils, when G = the specific gravity,

$$e = \frac{w}{100} \times G \quad \text{(1)}$$

This is because

$$e = \frac{\text{cubic centimeters of water}}{\text{cubic centimeters of solids}} \text{ and } w = \frac{\text{grams of water}}{\text{grams of solids}} \times 100$$

One cubic centimeter of water weighs 1 g. and 1 cu. cm. of solids weighs G g.

Therefore

$$w = \frac{\text{cubic centimeters of water}}{G \times \text{cubic centimeters of solids}} \times 100$$

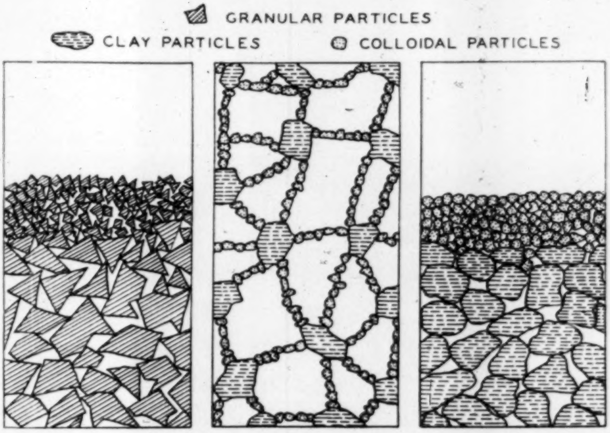
When $G = 2.71$ as in the case of the muck soil, sample No. 8170, used for illustrative purposes in the following discussion,

$$e = \frac{2.71\, w}{100} = 0.0271\, w$$

Test Results Indicate Density of Soils at Different Consistencies:

The significance of the routine soil test results may be excellently depicted by the performance of earth layers beginning when the soil particles first settle out of suspension, continuing while the sediments thus formed are drying or are being otherwise consolidated to constant weight and concluding with the rewetting of the dried soil.

During sedimentation, consolidation and shrinkage, some soil materials, such as sand, change but little, while others, like the colloidal clays, change enormously in density as well as stability and compressibility, which are mathematically related to density. The total change in density, together with the density at particularly selected degrees of shrinkage, swell, consolidation or expansion, indicates the extent to which soils have the five characterizing properties. Consequently, such densities are the basis of soil identification by means of the routine tests. Shrinkage tests disclose the variation in density; flow curves, stability; and the compression test, bearing value of the confined sample.



(a) GRANULAR SOIL **(b)** FLOCCULATED SOIL **(C)** SEDIMENT OF DEFLOCCULATED SOIL

Fig. 6.—Types of Soil Sediments.

The characterizing performance of soils is first noticed when the materials are forming sediments(11). The granular particles settle out of suspension independently and form stratified layers with the faster falling large grains on the bottom, as shown in Fig. 6 (*a*). In contrast with this, flocculated masses of colloids trap the larger particles and prevent independent movement when colloidal clays settle out of suspension. As a result the clay particles are distributed throughout the highly porous sediments thus formed without stratification, as illustrated in Fig. 6 (*b*).

Deflocculents, which destroy the electrolytic bonds of colloidal flocs, release the clay particles to settle individually like bulky grains and form stratified sediments as shown in Fig. 6 (*c*).

Figure 7 shows a flocculated layer to the left. It is uniform in appearance and has a moisture content of 130 per cent. The deflocculated sediment on the right is stratified and has a moisture content of 59 per cent. Both were formed in 24 hr. from suspensions of the same soil material. The one on the left was formed from 5 cu. cm. of solids thoroughly mixed with 40 ml. of distilled water. The one on the right was formed from 5 cu. cm. of solids thoroughly mixed with 39 ml. of distilled water and 1 ml. of deflocculent. The moisture contents of sediments formed in the latter manner are referred to below as "flocculation limits."

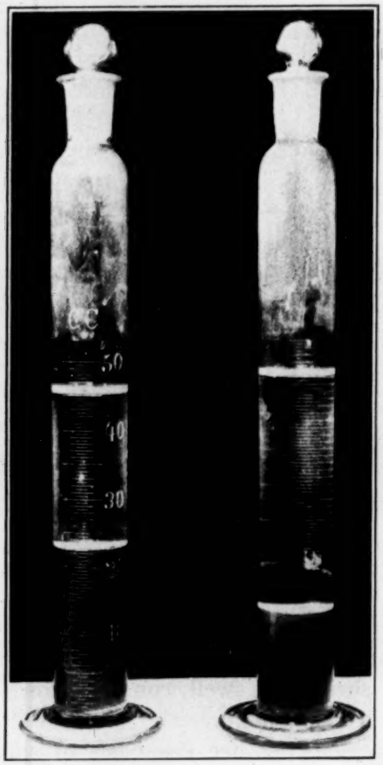
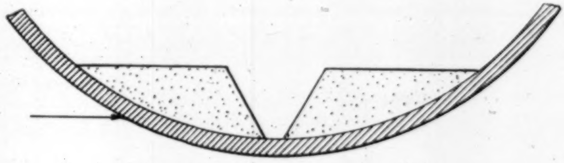


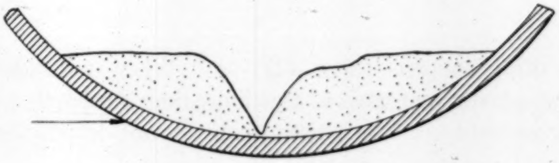
FIG. 7.—Effect of Flocculation on the Character of Soil Sediments.

STATES OF CONSISTENCY OF SOILS

Sediments of cohesive soils may be gradually consolidated from the liquid or viscous state at the flocculation limit to approximately the liquid limit by weight of the soil particles alone. Shrinkage or additional load may then cause the soil to pass successively the liquid limit, which is the upper limit of the plastic state; the plastic limit, which is the lower limit of the plastic state and the upper limit of the semi-solid state; and finally the shrinkage limit, which is the lower limit of the semi-solid state and the upper limit of the solid state.

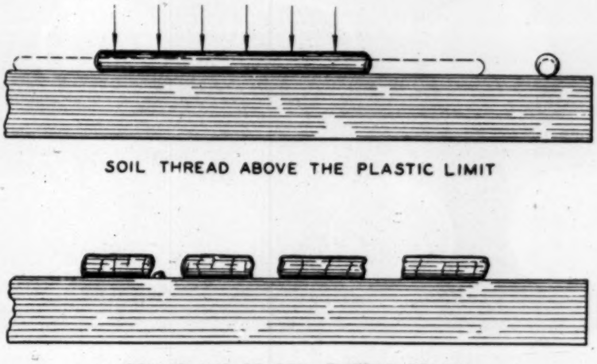


DIVIDED SOIL CAKE BEFORE TEST

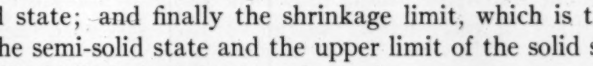


SOIL CAKE AFTER TEST

FIG. 8.--Phenomenon Occurring During the Liquid Limit Test.



SOIL THREAD ABOVE THE PLASTIC LIMIT



CRUMBLING OF SOIL THREAD BELOW THE PLASTIC LIMIT

FIG. 9.—Phenomenon Occurring During the Plastic Limit Test.

The liquid limit is defined as the moisture content at which ten light shocks will just close the groove in a soil sample such as shown in Fig. 8. Samples from the under soil at Four Mile Run(15) substantiate Terzaghi's statement of several years ago(16) that liquid limits approximately represent the natural moisture content of old sediments (see Table I).

The plastic limit is defined as the lowest moisture content at which the soil can be rolled into threads $\frac{1}{8}$ in. in diameter without the threads breaking to pieces (see Fig. 9). When soils change from plastic to non-plastic materials at this limit their supporting properties increase considerably. Non-plastic soils do not have plastic limits nor do they exist in plastic states.

TABLE I.—COMPARISON OF LIQUID LIMITS WITH MOISTURE CONTENTS OF SOIL IN NATURAL STATE. From *Public Roads*, March, 1934.

Boring	Moisture Content, per cent	
	Natural State	Liquid Limit
S-2	76	77
S-4	108	103
S-6	125	132
S-7	105	96
S-7	116	112
S-9	79	62

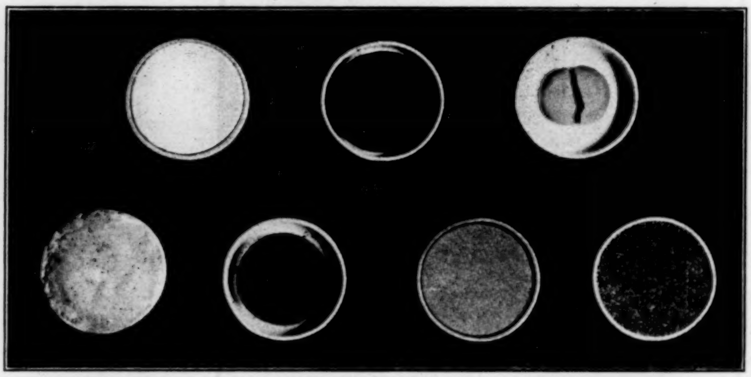


FIG. 10.—Illustrating the Shrinkage of Different Soil Materials when Dried to Constant Weight from Slightly Above the Liquid Limit.

The wet soil completely filled the Coors porcelain dishes at the beginning of the test. Top row, left to right: diatoms, peat, colloids. Bottom row: mica, clay, silt, sand.

The numerical difference between the liquid and plastic limits, defined as the plasticity index, shows the range in moisture contents through which the soil remains plastic. It increases with the cohesion of soils.

The shrinkage limit is defined as the moisture content at which further evaporation of moisture does not produce further decrease in the volume of the soil.

In the shrinkage test, soil powder and water are mixed into a paste slightly above the liquid limit, placed in a porcelain milk dish, as shown in Fig. 10, and then dried to constant weight. While drying, the sample loses volume proportionately to the moisture loss above the shrinkage limit as shown in Fig. 11. Here the volume change equals 335 per cent when the moisture content is reduced from 269 to 33 per cent. Consequently, the ratio of volume change to moisture loss, termed the "shrinkage ratio" (Fig. 11) equals:

$$\frac{335}{269 - 33} = \frac{335}{236} = 1.42$$

The shrinkage limit, the shrinkage ratio and the specific gravity are interrelated as follows:

$$\text{Shrinkage limit, } S = w - \frac{V - V_o}{W_o} \times 100 \qquad (2)$$

$$\text{Shrinkage ratio, } R = \frac{W_o}{V_o} \qquad (3)$$

$$\text{Specific gravity, } G = \frac{1}{\frac{1}{R} - \frac{S}{100}} \qquad (4)$$

where S = shrinkage limit, moisture content, per cent,
w = moisture content, of wet soil, per cent,
V = volume of wet-soil cake, cubic centimeters,
V_o = volume of dry-soil cake, cubic centimeters,
W = weight of wet-soil cake, grams,
W_o = weight of dry-soil cake, grams,
R = shrinkage ratio, and
G = specific gravity of soil particles.

The volume change C_o which occurs when a soil sample is dried from any moisture content w is given by the equation:

$$C_o = (w - S)\,R \qquad (5)$$

$$= \frac{w - S}{\frac{1}{G} + \frac{S}{100}}$$

This for sample No. 8170 becomes:

$$C_o = (w - 33)\,1.42$$

The shrinkage ratio R is also the apparent specific gravity of the soil. Thus the weight of a cubic foot of the muck in the dried state equals 62.5 × 1.42 = 89 lb.

The volumetric change is defined as the volume change in percentage of the dry volume which occurs when the moisture content of the sample is reduced from the field moisture equivalent to 0. It is computed from Eq. 5 by substitution of the field moisture equivalent, F.M.E., for w. The volumetric change when designated by C_f becomes:

$$C_f = (\text{F.M.E.} - S)R \quad \ldots\ldots (6)$$

The lineal shrinkage, L.S.(17), expressed as a percentage of the length of a wet soil sample, is defined by the equation:

$$\text{L.S.} = 100\left[1 - \sqrt[3]{\frac{100}{C_f + 100}}\right] \quad \ldots\ldots (7)$$

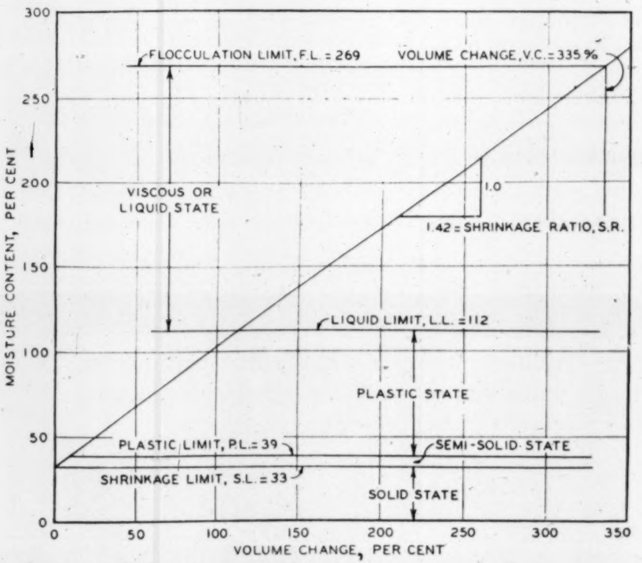


FIG. 11.—Relationship of Volume Change to Moisture Content at Different States of Consistency. Soil sample No. 8170.

The relative volumes of sample No. 8170 at the different limits of consistency are as follows:

Consistency Limit	Moisture Content, per cent	Volume Change When Dried to Constant Weight, Eq. 5, per cent	Relative Volume, percentage of volume of dried soil
Flocculation	269	335	435
Liquid	112	112	212
Plastic	39	7	107
Shrinkage	33	0	100

Thus 100 cu. ft. of the muck soil at the flocculation limit becomes 49 cu. ft. at the liquid ($\frac{212}{485} \times 100$); 25 cu. ft. at the plastic; and 23 cu. ft. at the shrinkage limit.

DETERMINATION OF FLOW CURVES

The apparatus illustrated in Fig. 12 was developed by Casagrande(13) for determining the liquid limit mechanically. The number of shocks required to close the groove in the soil at different consistencies, plotted against the moisture contents, form the flow curves. They are straight lines on semilogarithmic plats and are expressed by:

$$F = \frac{H - w}{\log N} \qquad (8)$$

where w is the moisture content at N shocks.

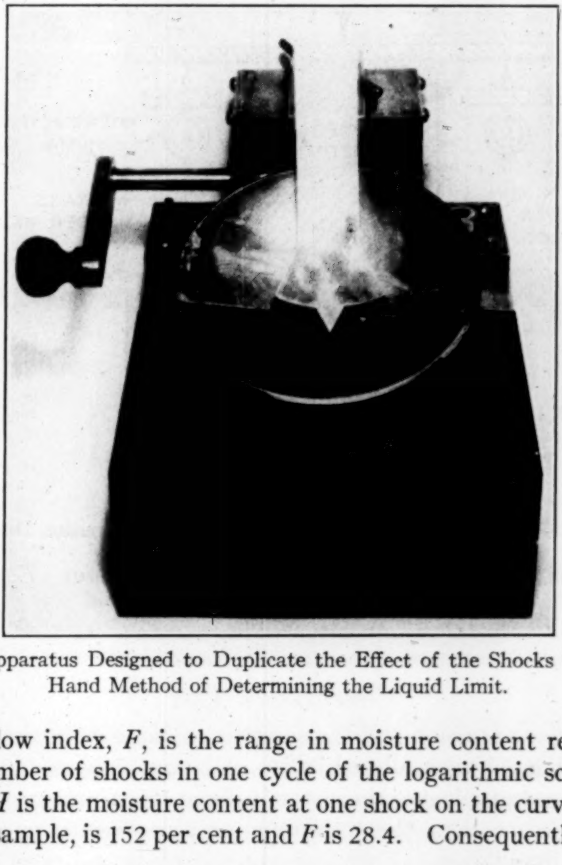


FIG. 12.—Apparatus Designed to Duplicate the Effect of the Shocks Used in the Hand Method of Determining the Liquid Limit.

The flow index, F, is the range in moisture content represented by the number of shocks in one cycle of the logarithmic scale. The intercept H is the moisture content at one shock on the curve. H, for the muck sample, is 152 per cent and F is 28.4. Consequently, for this sample

$$w = 152 - 28.4 \log N$$

The liquid limit is the moisture content at 25 shocks on the flow curve as compared with 10 shocks by the hand method. The mechanical device could be adjusted so that 10 shocks would indicate the liquid limit on the flow curves also. However, this would only increase the wear and tear on the apparatus without benefiting the test.

On the assumption that the shocks required to close the groove are an index of shear resistance, the flow curves indicate the influence of change of moisture content upon the stability of soils. Also, if the shocks at the plastic limit on the flow curve can be taken as a measure of the toughness of soil, the number of logarithmic cycles included within the range of moisture contents comprising the plasticity index, I, on the flow curve can be used as a toughness index, T. It is expressed as

$$T = \frac{I}{F} \tag{9}$$

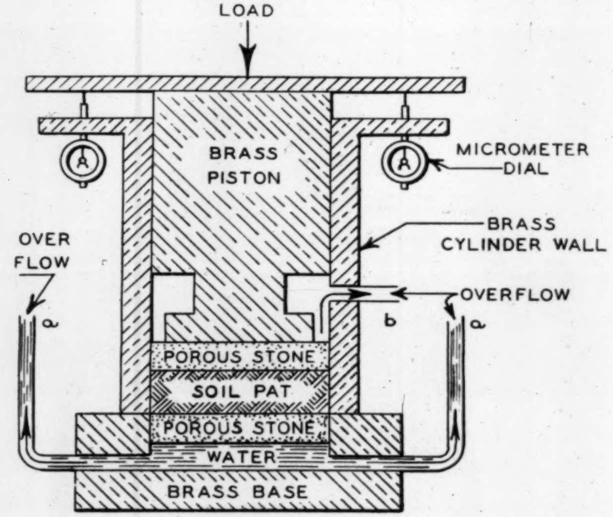


FIG. 13.—Essential Elements of the Terzaghi Compression Device.

COMPRESSION TEST

In the compression test the samples are encased in the apparatus shown in Fig. 13 with all possibility of lateral flow excluded (6,8,12,18). Static loads are applied to the piston in about three increments, totaling somewhat more than the maximum unit support required by the particular foundation. Each increment is continued until no further consolidation of the sample occurs. Then the next increment is added. After the sample has been compressed to constant volume under the final increment, the load is reduced to zero by decrements equal to the increments comprising the total load.

Water pressed from the soil pat by the weighed piston passes through the porous stone filters above and below the pat and escapes from the overflow orifices *a* and *b*.

The test furnishes the data shown in Fig. 14. The load-compression curve shows the voids ratio at which the sample will support a given load. The load-expansion curve shows the relation of increase in voids ratio to decrease of load. The time-compression curve shows the speed at which the test sample consolidates.

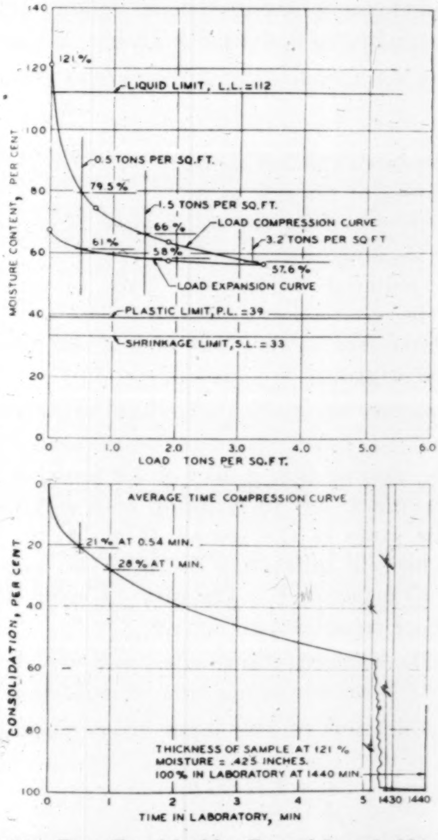


Fig. 14.—Data Furnished by Test of Sample No. 8170.

According to the load-compression curve, a load of 0.5 tons per sq. ft. compresses the sample No. 8170 to a moisture content of 79.5 per cent. According to the load-expansion curve, decreasing the load from 1.5 tons per sq. ft. to 0.5 ton per sq. ft. increases the moisture content from 58 per cent to 61 per cent. According to the time-compression curve 28 per cent of the consolidation occurs in 1 min.

Computation of Rate of Settlement of Soil Strata:

The relative periods of load application producing equal percentages of compression in soil strata and their representative laboratory samples are expressed by equations as follows:

Case 1.—Water free to flow from both top and bottom of the soil stratum:

$$\frac{t_a}{t_f} = \frac{a^2}{f^2} \qquad (10)$$

where a = thickness of the soil sample,

f = thickness of the soil layer,

t_a = time required for the soil sample to consolidate from w_o to w_1, and

t_f = time required for the soil layer to consolidate from w_o to w_1.

Case 2.—Water free to flow out of but one face, either top or bottom of the soil stratum:

$$\frac{t_a}{t_f} = \frac{a^2}{4f^2} \qquad (11)$$

To illustrate, assume that a load of 3.2 tons per sq. ft. is placed on a layer of the muck soil 10 ft. thick with a moisture content of 121 per cent, the same as the sample (Fig. 14) at the start of the loading test. This will ultimately decrease the moisture content of the sediment to 57.6 per cent. The ratio of layer thickness after to the thickness before consolidation equals:

$$\frac{100 + \text{volume change after consolidation}}{100 + \text{volume change before consolidation}}$$

Hence the thickness of the layer after ultimate consolidation (see Eq. 5) is

$$10 \times \frac{100 + 1.42\ (57.6 - 33)}{100 + 1.42\ (121 - 33)} = 6.0 \text{ ft.}$$

The test sample at 121 per cent of moisture is 0.425 in. thick according to the test data.

At some stage of partial consolidation, say 30 days after the load was applied, the moisture content of the soil layer would be reduced the same amount as that of the test sample in

$$t_a = \frac{30 \times 1440 \times (0.425)^2}{(12 \times 10)^2} = 0.54 \text{ min. (Eq. 10)}$$

The time-compression curve, Fig. 14, indicates that 21 per cent of the consolidation has occurred in this time. Consequently, the thickness of the muck deposit 30 days after the application of the 3.2 tons per sq. ft. load is

$$10 - \frac{21}{100}(10 - 6.0) = 9.2 \text{ ft.}$$

The load-compression curves within the range from the plastic limit to a very soft consistency ($P < 0.1$ ton per sq. ft.) are like the flow curves, straight lines on semi-logarithmic plats and have the form

$$w = B - S \log P \quad \text{(12)}$$

in which B is the moisture content corresponding to the 1 ton per sq. ft. load and S is the slope of the semi-logarithmic plat which may be termed also the compression index of the soil. It equals the variation in moisture content corresponding to a variation in load represented by one cycle on the semi-logarithmic scale.

For the muck sample the formula becomes:

$$w = 71 - 28.4 \log P$$

Combining Eq. 5 for volume change, Eq. 8 for the flow curve and Eq. 12 for compression, and substituting the constants of the muck sample, furnishes the expression:

$$w = \frac{C_o}{1.42} + 33 = 152 - 28.4 \log N = 71 - 28.4 \log P$$

Therefore, volume change, shocks and bearing value corresponding to any desired moisture content above the plastic limit may be determined. These for sample No. 8170 at three arbitrarily chosen moisture contents are as follows:

w, PER CENT	C_o, PER CENT	N, SHOCKS	P, TONS PER SQ. FT.
100	95	68	0.097
75	60	513	0.74
50	24	3888	5.55

The average increase in moisture per decrease of 1 ton per sq. ft. when the total load is reduced from about 3 tons per sq. ft. to 0 may be used to signify the relative expansion properties of soil. For the muck sample this expansion factor equals 3.0 per cent.

MOISTURE EQUIVALENT TESTS

The moisture equivalent tests indicate compressive and expansive properties.

The centrifuge moisture equivalent is defined as the moisture content of the soil when compressed by a centrifugal force of 1000 times the force of gravity for 1 hr. Essentially, the test (see Fig. 15) consists of first soaking a small sample of air-dried soil in a Gooch crucible, then draining it in a humidifier for at least 12 hr., and finally centrifuging it for 1 hr.

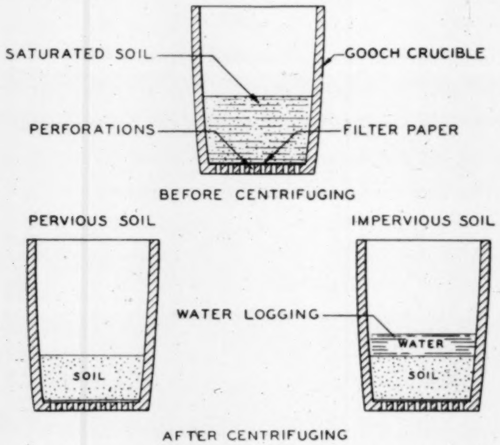

SATURATED SOIL
GOOCH CRUCIBLE
PERFORATIONS
FILTER PAPER
BEFORE CENTRIFUGING
PERVIOUS SOIL
IMPERVIOUS SOIL
WATER LOGGING
WATER
SOIL
SOIL
AFTER CENTRIFUGING

FIG. 15.—Phenomenon Occurring During the Centrifuge Moisture Equivalent Test.

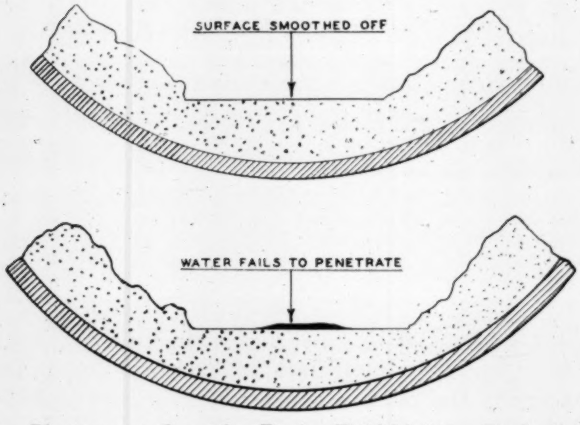
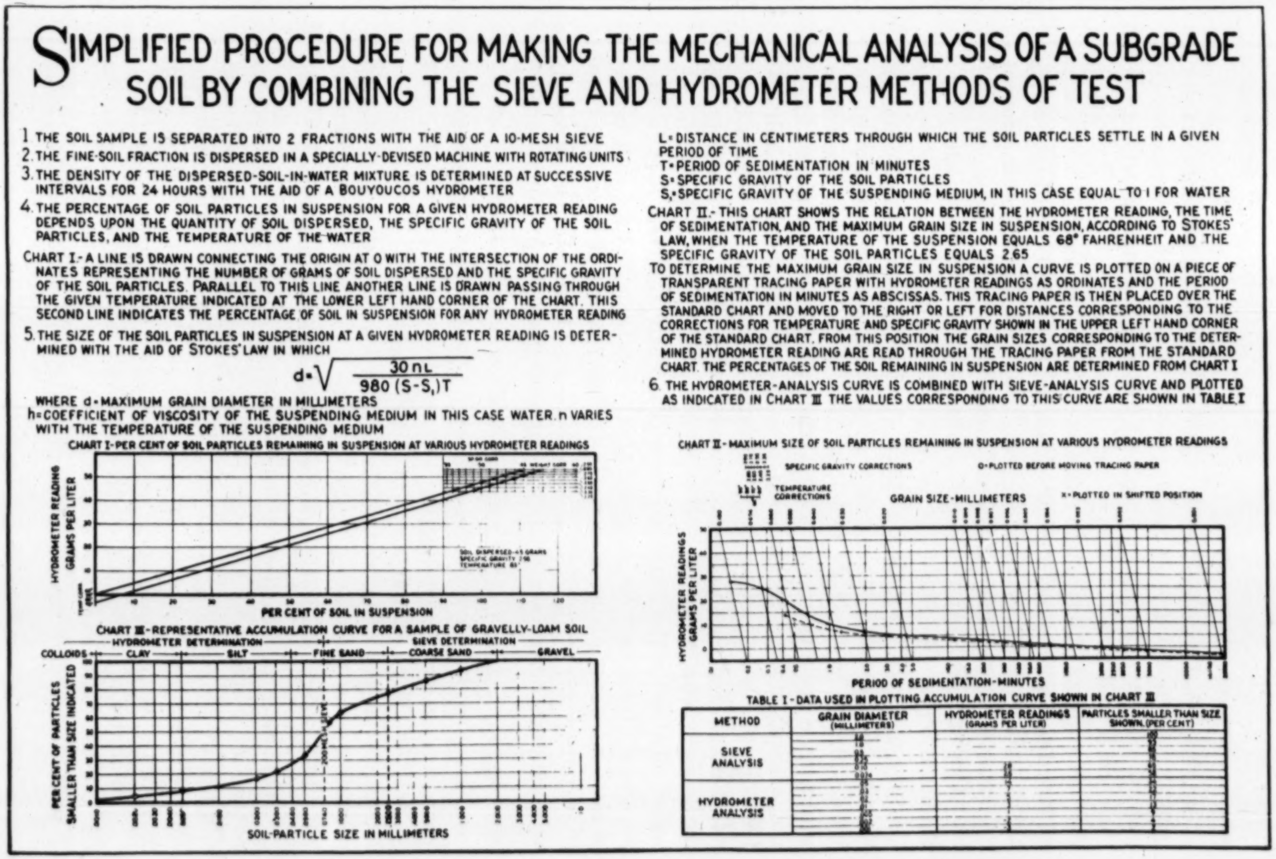

SURFACE SMOOTHED OFF
WATER FAILS TO PENETRATE

FIG. 16.—Phenomenon Occurring During Field Moisture Equivalent Test.

The centrifugal action has a two-fold effect: namely, (*a*) to consolidate the soil particles forcing water toward both top and bottom of the sample, and (*b*) to expel water through the perforations in the bottom of the crucible. This combination of forces produces an average pressure on the sample of about 2 kg. per sq. cm. All free water is expelled from pervious samples. At least part of the water



SIMPLIFIED PROCEDURE FOR MAKING THE MECHANICAL ANALYSIS OF A SUBGRADE SOIL BY COMBINING THE SIEVE AND HYDROMETER METHODS OF TEST

1 THE SOIL SAMPLE IS SEPARATED INTO 2 FRACTIONS WITH THE AID OF A 10-MESH SIEVE

2. THE FINE-SOIL FRACTION IS DISPERSED IN A SPECIALLY-DEVISED MACHINE WITH ROTATING UNITS

3. THE DENSITY OF THE DISPERSED-SOIL-IN-WATER MIXTURE IS DETERMINED AT SUCCESSIVE INTERVALS FOR 24 HOURS WITH THE AID OF A BOUYOUCOS HYDROMETER

4. THE PERCENTAGE OF SOIL PARTICLES IN SUSPENSION FOR A GIVEN HYDROMETER READING DEPENDS UPON THE QUANTITY OF SOIL DISPERSED, THE SPECIFIC GRAVITY OF THE SOIL PARTICLES, AND THE TEMPERATURE OF THE WATER

CHART I.- A LINE IS DRAWN CONNECTING THE ORIGIN AT O WITH THE INTERSECTION OF THE ORDINATES REPRESENTING THE NUMBER OF GRAMS OF SOIL DISPERSED AND THE SPECIFIC GRAVITY OF THE SOIL PARTICLES. PARALLEL TO THIS LINE ANOTHER LINE IS DRAWN PASSING THROUGH THE GIVEN TEMPERATURE INDICATED AT THE LOWER LEFT HAND CORNER OF THE CHART. THIS SECOND LINE INDICATES THE PERCENTAGE OF SOIL IN SUSPENSION FOR ANY HYDROMETER READING

5. THE SIZE OF THE SOIL PARTICLES IN SUSPENSION AT A GIVEN HYDROMETER READING IS DETERMINED WITH THE AID OF STOKES' LAW IN WHICH

$$d = \sqrt{\frac{30 n L}{980 (S - S_1) T}}$$

WHERE d = MAXIMUM GRAIN DIAMETER IN MILLIMETERS

h = COEFFICIENT OF VISCOSITY OF THE SUSPENDING MEDIUM IN THIS CASE WATER. n VARIES WITH THE TEMPERATURE OF THE SUSPENDING MEDIUM

L = DISTANCE IN CENTIMETERS THROUGH WHICH THE SOIL PARTICLES SETTLE IN A GIVEN PERIOD OF TIME

T = PERIOD OF SEDIMENTATION IN MINUTES

S = SPECIFIC GRAVITY OF THE SOIL PARTICLES

S_1 = SPECIFIC GRAVITY OF THE SUSPENDING MEDIUM, IN THIS CASE EQUAL TO 1 FOR WATER

CHART II.- THIS CHART SHOWS THE RELATION BETWEEN THE HYDROMETER READING, THE TIME OF SEDIMENTATION, AND THE MAXIMUM GRAIN SIZE IN SUSPENSION, ACCORDING TO STOKES' LAW, WHEN THE TEMPERATURE OF THE SUSPENSION EQUALS 68° FAHRENHEIT AND THE SPECIFIC GRAVITY OF THE SOIL PARTICLES EQUALS 2.65

TO DETERMINE THE MAXIMUM GRAIN SIZE IN SUSPENSION A CURVE IS PLOTTED ON A PIECE OF TRANSPARENT TRACING PAPER WITH HYDROMETER READINGS AS ORDINATES AND THE PERIOD OF SEDIMENTATION IN MINUTES AS ABSCISSAS. THIS TRACING PAPER IS THEN PLACED OVER THE STANDARD CHART AND MOVED TO THE RIGHT OR LEFT FOR DISTANCES CORRESPONDING TO THE CORRECTIONS FOR TEMPERATURE AND SPECIFIC GRAVITY SHOWN IN THE UPPER LEFT HAND CORNER OF THE STANDARD CHART. FROM THIS POSITION THE GRAIN SIZES CORRESPONDING TO THE DETERMINED HYDROMETER READING ARE READ THROUGH THE TRACING PAPER FROM THE STANDARD CHART. THE PERCENTAGES OF THE SOIL REMAINING IN SUSPENSION ARE DETERMINED FROM CHART I

6. THE HYDROMETER-ANALYSIS CURVE IS COMBINED WITH SIEVE-ANALYSIS CURVE AND PLOTTED AS INDICATED IN CHART III THE VALUES CORRESPONDING TO THIS CURVE ARE SHOWN IN TABLE I

CHART I - PER CENT OF SOIL PARTICLES REMAINING IN SUSPENSION AT VARIOUS HYDROMETER READINGS

HYDROMETER READING GRAMS PER LITER
PER CENT OF SOIL IN SUSPENSION
SP GR CORR
WEIGHT CORR
SOIL DISPERSED-45 GRAMS
SPECIFIC GRAVITY 2.66
TEMPERATURE 81°
TEMP CORR

CHART III - REPRESENTATIVE ACCUMULATION CURVE FOR A SAMPLE OF GRAVELLY-LOAM SOIL

HYDROMETER DETERMINATION
SIEVE DETERMINATION
COLLOIDS
CLAY
SILT
FINE SAND
COARSE SAND
GRAVEL
200-MESH SIEVE
PER CENT OF PARTICLES SMALLER THAN SIZE INDICATED
SOIL-PARTICLE SIZE IN MILLIMETERS

CHART II - MAXIMUM SIZE OF SOIL PARTICLES REMAINING IN SUSPENSION AT VARIOUS HYDROMETER READINGS

SPECIFIC GRAVITY CORRECTIONS
TEMPERATURE CORRECTIONS
O = PLOTTED BEFORE MOVING TRACING PAPER
X = PLOTTED IN SHIFTED POSITION
GRAIN SIZE-MILLIMETERS
HYDROMETER READINGS GRAMS PER LITER
PERIOD OF SEDIMENTATION-MINUTES

TABLE I - DATA USED IN PLOTTING ACCUMULATION CURVE SHOWN IN CHART III

METHOD	GRAIN DIAMETER (MILLIMETERS)	HYDROMETER READINGS (GRAMS PER LITER)	PARTICLES SMALLER THAN SIZE SHOWN (PER CENT)
SIEVE ANALYSIS	[illegible]	[illegible]	[illegible]
HYDROMETER ANALYSIS	[illegible]	[illegible]	[illegible]

FIG. 17.—Simplified Procedure for Making the Mechanical Analysis of Subgrade Soil.

forced to the top of impervious samples is retained there to produce what is termed "waterlogging."

Field moisture equivalent is defined as the minimum moisture content at which a drop of water placed on a smoothed surface of the soil will not immediately be absorbed, but will instead spread out over the surface and give it a shiny appearance (see Fig. 16). Essentially the test consists of thoroughly mixing small increments of water with air-dried soil and smoothing off with a spatula until additional water, as referred to above, will not be absorbed. At this moisture content, the expansive properties of the soil sample have been satisfied.

The specific gravities are determined by a method which is a modification of the Society's Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (D 70 – 27).[1] It consists of placing a known weight of air-dried soil, the hygroscopic moisture content of which has been determined, in a specific gravity bottle having a perforated glass stopper and adding enough distilled water to fill the bottle about half full. The soil and water mixture is gently boiled for 5 min., the bottle being rolled occasionally to assist in the removal of air. After boiling, the bottle and contents are allowed to cool, the bottle is filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for a period of not less than 30 min. in a beaker of distilled water maintained at 25 C., after which it is removed and all surplus water is wiped off with a clean dry cloth. It is immediately weighed and the specific gravity computed from the formula:

$$\text{Specific gravity} = \frac{c - a}{(b - a) - (d - c)}$$

where a = weight of dry bottle,
b = weight of bottle full of distilled water,
c = weight of bottle plus weight of air-dry soil minus weight of hygroscopic moisture, and
d = weight of bottle filled with soil and distilled water.

The essential features of the combined sieve and hydrometer method of mechanical analysis, including the graphical method of solution of Stokes' law, are shown in Fig. 17(19, 20). How substantially the results furnished by the hydrometer method agree with those of the more cumbersome pipette method is shown in Table II.

In addition to the particle size accumulation curves, the effective size and the uniformity coefficient may be used to indicate the grading of soils. The effective size(21) is the maximum size of the smallest

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[1] 1933 Book of A.S.T.M. Standards, Part II, p. 1002.

10 per cent, by weight, of the soil particles. The abscissa of the point having an ordinate of 10 per cent on the soil accumulation curve (see Fig. 17) discloses the effective size. The uniformity coefficient (21) is the effective size divided into the maximum size of the smallest 60 per cent, by weight, of the soil particles.

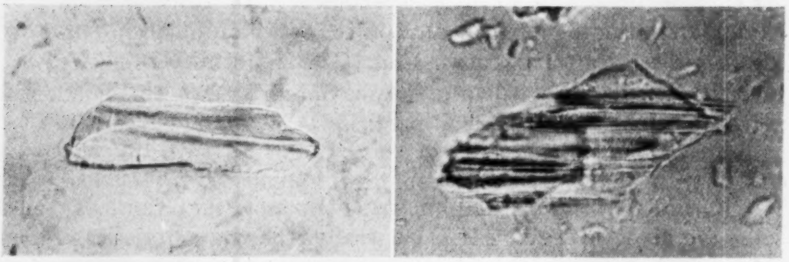
ILLUSTRATIONS OF VARIATION IN THE PHYSICAL PROPERTIES OF SOILS

Results of tests on well-known commercial materials, as well as soils, are given in Table III. Generally, the characteristics of these materials include all those found in natural soils. Bentonite, at the top of the list, is the most active of the gluey colloids productive of compressibility, plasticity and shrinkage of soils. Ground quartz, at the bottom, is representative of the most stable of cohesionless fine-grained materials of more or less constant volume.

TABLE II.—PERCENTAGES OF THE FINER SOIL PARTICLES AS DETERMINED BY THE BOUYOUCOS HYDROMETER AND THE PIPETTE METHOD.

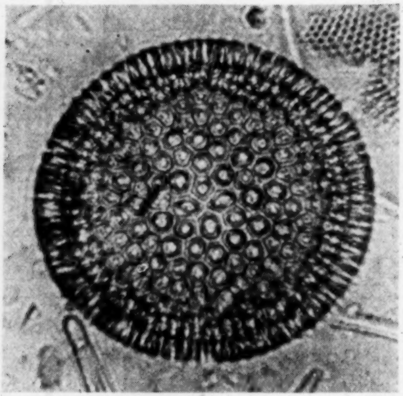
Sample	Percentage of Particles Finer Than					
	0.05 mm.		0.005 mm.		0.002 mm.	
	Pipette Method	Bouyoucos Hydrometer	Pipette Method	Bouyoucos Hydrometer	Pipette Method	Bouyoucos Hydrometer
No. 802	99	100	78	78	54	57
No. 4295	95	97	38	32	29	28
No. 837	94	95	75	72	64	62
No. 872	88	89	45	41	26	29
No. 695	83	85	47	48	36	38
No. 630	81	83	40	41	27	26
No. 4258	81	85	21	22	14	11
No. 659	77	79	22	21	15	15
No. 763	69	67	36	38	26	30
No. 4913	59	56	24	27	14	19
No. 487	58	57	18	16	14	11
No. 4907	12	13	8	9	7	4

Zinc oxide, the finest material in the group, is representative of the relatively inert clays in glacial formations which are responsible for enormous frost heave. Diatoms and mica with little or no cohesion, high capillarity and high shrinkage limits, are productive of elasticity of soils. Pumice, with no cohesion and a fairly constant and high moisture content at the several consistencies, is representative of those quicksands which appear dry on the surface but quake and flow at the slightest manipulation. Sample No. 7588 is typical of clay soils found in nature. The addition of diatoms and peat to such soils produces river bottom mucks having the properties of sample No. 8170. Kaolin is representative of good pottery clays, which also make excellent binders for topsoil, sand-clay and gravel road surfaces. Talc has the physical properties of the natural cohesionless silt soils noted for the occurrence of detrimental frost heave.

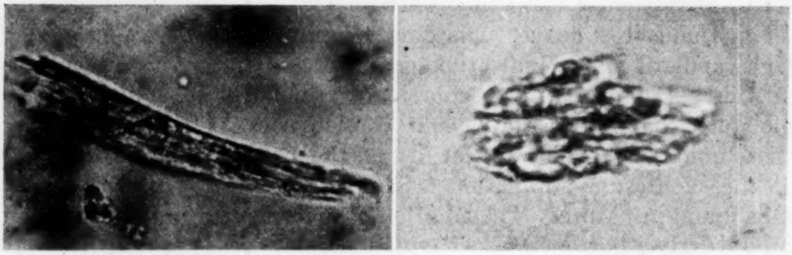


Talc
Relative magnification, 1

Pumice
Relative magnification, 1



Diatoms
Relative magnification, 2.2



Peat
Relative magnification, 2.2

Bentonite
Relative magnification, 3.8

FIG. 18.—Photomicrographs of Soil Materials.

The widely different grain structure of several of the materials shown in Fig. 18 adequately explains the difference in performance. Amorphous organic matter and a diatom can be seen in Fig. 19 of a sample of the muck soil No. 8170.

The great range of characteristics of these materials is noted as follows:

1. Maximum moisture content indicated by the flocculation limit −46 to more than 1000 per cent.

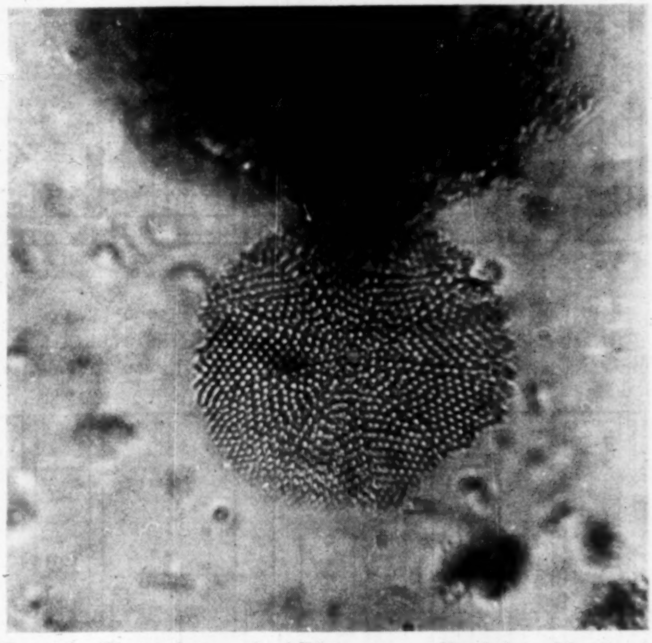


FIG. 19.—Photomicrograph of Diatomaceous Earth from Maryland.

2. Moisture content at which the materials have equal shear resistance, that indicated by 25 blows signifying the liquid limit 18 to 399 per cent.
3. Cohesion indicated by the plasticity index −0 to 354 per cent.
4. Shrinkage properties as indicated by the volume change from the liquid limit −0 to 865 per cent.
5. Relative effect of moisture increase for reducing the shear resistance of the soil as indicated by the flow index −4 to 61 per cent.
6. Moisture content at which the samples in confined state are capable of supporting a load equal to 1 ton per sq. ft. as indicated by the compression intercept, B − 23 to 221 per cent.

7. The relative ability of the materials to absorb water and expand upon the removal of load as indicated by the expansion factor —0.2 to 21.9 per cent.

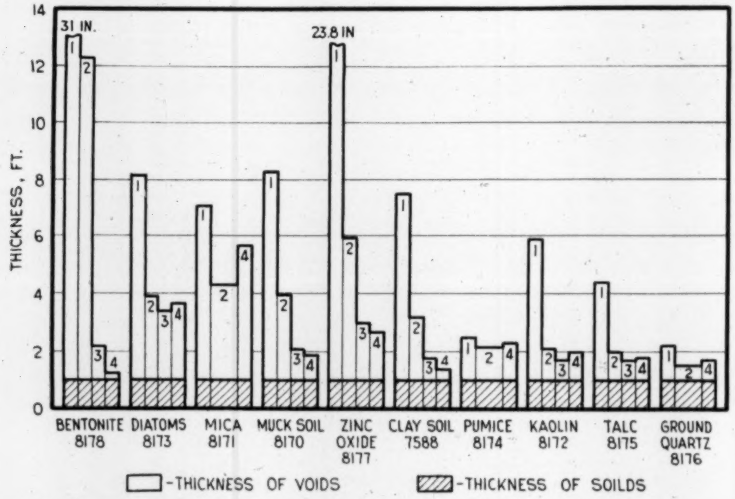


FIG. 20.—Relative thickness of layers of materials listed in Table III at flocculation limit (1), liquid limit (2), plastic limit (3), and shrinkage limit (4).

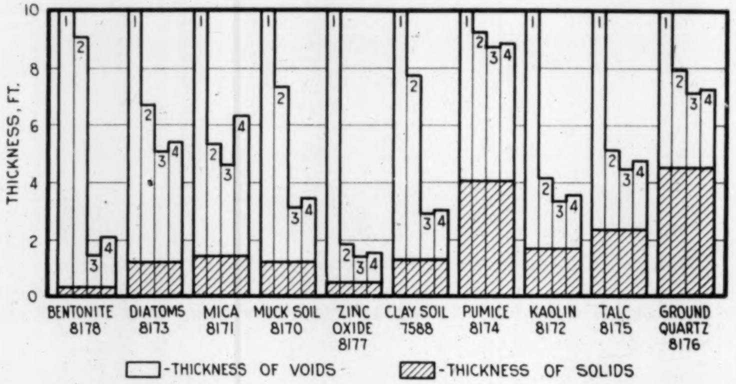


FIG. 21.—Relative thickness of layers of materials listed in Table III when a layer 10 ft. thick at the flocculation limit (1), compressed by a load of approximately 3 tons per sq. ft. for 30 days (2), to ultimate consolidation or constant volume (3), and when the layer expands to constant volume upon the removal of load (4).

Figures 20 and 21 illustrate how differences in physical character affect the volume of soils. Figure 20 shows the relative thicknesses of the materials, Table III, at the different consistency limits, with a constant thickness of solids equal to 1 ft. Figure 21 shows the effect

TABLE III.—TEST CONSTANTS OF TYPICAL FINE-GRAINED MATERIALS.

Material	Grading		Consistency				Shrinkage		Volume Change from Liquid Limit, per cent	Flow Curve			Compression			Specific Gravity
	Effective Size, mm.	Uniformity Coefficient	Flocculation Limit, per cent	Liquid Limit, per cent	Plastic Limit, per cent	Plasticity Index	Limit, per cent	Ratio		Flow Index, F, per cent	Intercept, H, per cent	Toughness Index	Compression Index, S, per cent	Intercept, B, per cent	Expansion Factor	
Bentonite	0.0002	0	1060	399	45	354	6	2.2	865	33.0	438	10.7	190.5	220.8	21.9	2.83
Diatoms	0.0003	9.1	337	136	115	21	125	0.6	7	31.7	181	0.7	24.0	159.4	4.3	2.12
Mica	0.0023	9.1	214	116	...	0	165[a]	0.6	0	44.0	176	0	68.1	113.0	13.0	2.85
Muck	0.0003	7.0	269	112	39	73	33	1.4	111	28.4	152	2.6	28.4	71.4	3.1	2.71
Zinc oxide	0.0001	2.4	408	89	35	54	31	1.9	110	61.0	147	0.9	48.3	68.0	0.2	5.59
Clay soil	0.0002	8.8	236	80	29	51	13	1.9	127	25.0	114	2.0	25.7	54.7	0.9	2.76
Pumice	0.0066	5.0	64	52	...	0	57[a]	1.0	0	4.5	58	0	5.3	52.5	0.4	2.34
Kaolin	0.0004	11.0	186	43	28	15	37	1.3	8	14.0	63	1.1	10.3	40.7	1.4	2.63
Talc	0.0028	7.5	123	36	25	11	29	1.4	10	18.6	61	0.6	12.3	40.4	1.3	2.77
Quartz flour	0.0021	19.1	46	18	...	0	26[a]	1.6	0	4.9	26	0	1.9	23.1	0.4	2.67

[a] Computed from shrinkage ratio and specific gravity.

TABLE IV.—MATERIALS IN TABLE III ARRANGED IN DESCENDING ORDER OF EXTENT OF FINENESS AND OTHER PROPERTIES.

Fineness	Flocculation Limit	Liquid Limit	Plasticity Index	Volume Change from Liquid Limit	Flow Index, F	Intercept, B	Expansion Factor
Zinc oxide	Bentonite	Bentonite	Bentonite	Bentonite	Zinc oxide	Bentonite	Bentonite
Bentonite	Zinc oxide	Diatoms	Muck	Clay	Mica	Diatoms	Mica
Clay	Diatoms	Mica	Zinc oxide	Muck	Bentonite	Mica	Diatoms
Muck	Muck	Muck	Clay	Zinc oxide	Diatoms	Muck	Muck
Diatoms	Clay	Zinc oxide	Diatoms	Talc	Muck	Zinc oxide	Kaolin
Kaolin	Mica	Clay	Kaolin	Kaolin	Clay	Clay	Talc
Quartz flour	Kaolin	Pumice	Talc	Diatoms	Talc	Pumice	Clay
Mica	Talc	Kaolin	Mica	Mica	Kaolin	Kaolin	Pumice
Talc	Pumice	Talc	Pumice	Pumice	Quartz flour	Talc	Quartz flour
Pumice	Quartz flour	Quartz flour	Quartz flour	Quartz flour	Pumice	Quartz flour	Zinc oxide

TABLE V.—EFFECT OF ADMIXTURES ON PLASTICITY TESTS.

Material, per cent			Liquid Limit	Plasticity Index
Ground Quartz	Diatoms	Bentonite		
100	0	....	18	0
80	20	....	40	2
60	40	....	61	5
40	60	....	86	15
20	80	....	112	17
0	100	....	136	21
100	....	0	18	0
95	....	5	32	16
85	....	15	61	45
75	....	25	96	79
60	....	40	153	133
0	....	100	399	354

of the relative rate and ultimate settlement under equal load, and the expansion upon removal of load, upon the thickness of a soil layer.

The inability of the mechanical analysis or any other one determination to throw light on all the properties is illustrated by Table IV. Here the materials, Table III, are listed with respect to their fineness and also the extent to which they possess the several physical properties listed above under Nos. 1 to 7, inclusive. In almost every column the arrangement is different, thus indicating that complete identification of the soil can be accomplished only by tests sufficient in number and varied enough in scope to disclose the extent to which all five dominating physical characteristics are present.

The routine tests reflect in some form or other the influence of the basic soil properties as follows:

Liquid Limit.—Capillary capacity of soil when thoroughly manipulated.

Plasticity Index.—Cohesion.

Shrinkage Limit.—Combined effect of cohesion and resistance to consolidation.

Centrifuge Moisture Equivalent.—Combined effect of capillarity, compression and permeability.

Field Moisture Equivalent.—Combined effect of capillarity and cohesion—the moisture capacity of soils when not manipulated.

Volumetric Change and Lineal Shrinkage.—Combined effect of capillarity, cohesion and resistance to consolidation.

Routine Tests Reflect the Presence of Characterizing Soil Materials:

The routine tests disclose basic soil properties by indicating the presence of constituents which, by surveys, investigations and experience, have been shown to have a characterizing influence upon the construction and maintenance of road surfaces. This is illustrated by tests on changing combinations of widely different materials such as quartz, diatomaceous earth and bentonite.

Both Table V and Fig. 22 show that increasing the amount of admixture gradually changes the constants from those of the stable quartz to those of the diatoms, in one instance, and the highly plastic colloidal bentonite in the other. Both the liquid limits and the plasticity indexes increase proportionately with the admixtures. Consequently, the plasticity indexes increase directly with the liquid limits but at different ratios for the different admixtures.

In like manner, all of the characterizing soil constituents such as sand, silt, clay, colloids, kaolin, mica, peat, the flocculating limes and magnesium and the deflocculating potash and sodas contribute individual properties indicated by test constants related in ratios covering a wide range. Such relationships supplement the constants as such in the identification of properties productive of conspicuous performance.

Based upon constituent materials, physical properties and performance, uniform subgrades are tentatively classified in eight groups, identified by definite test limits and requiring different construction features in roads laid thereon. This grouping is illustrated by Fig. 23.

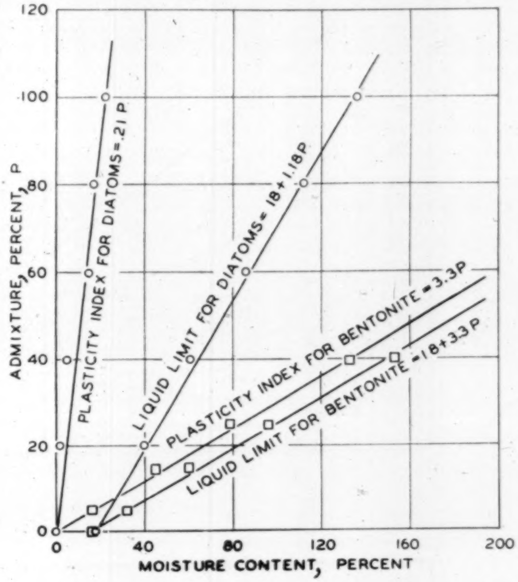


Fig. 22.—Relationship of Liquid Limits and Plasticity Indexes to Admixtures of Diatoms and Bentonite.

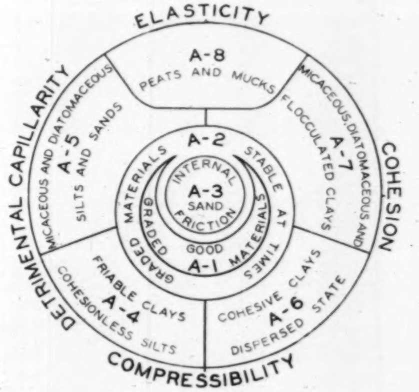
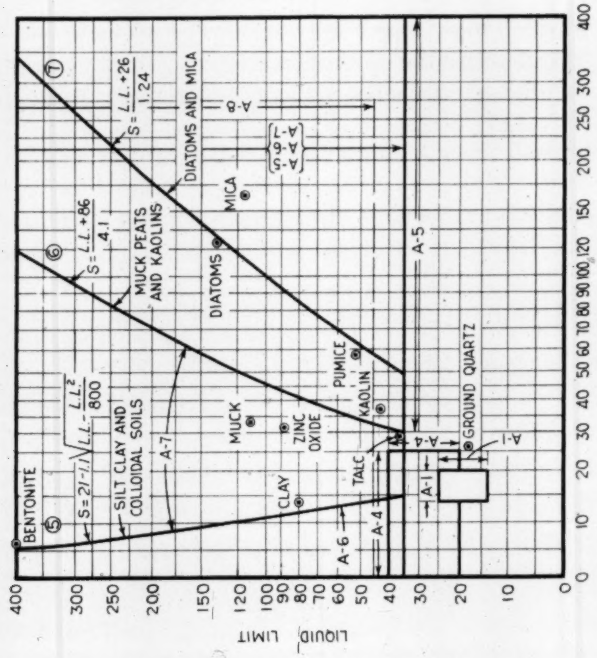
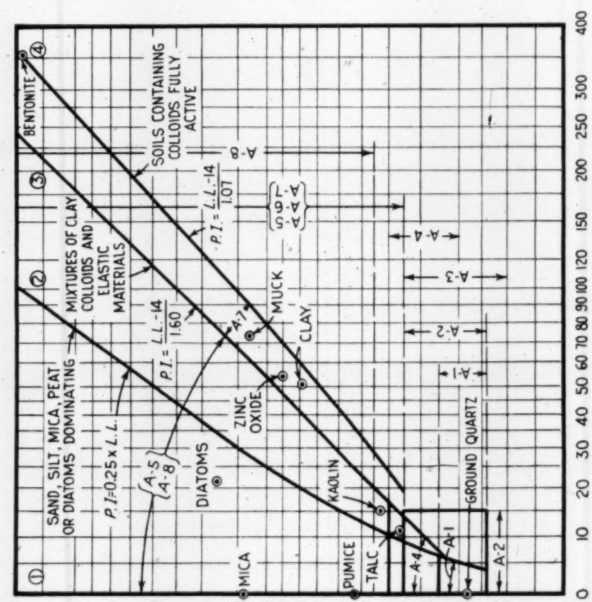
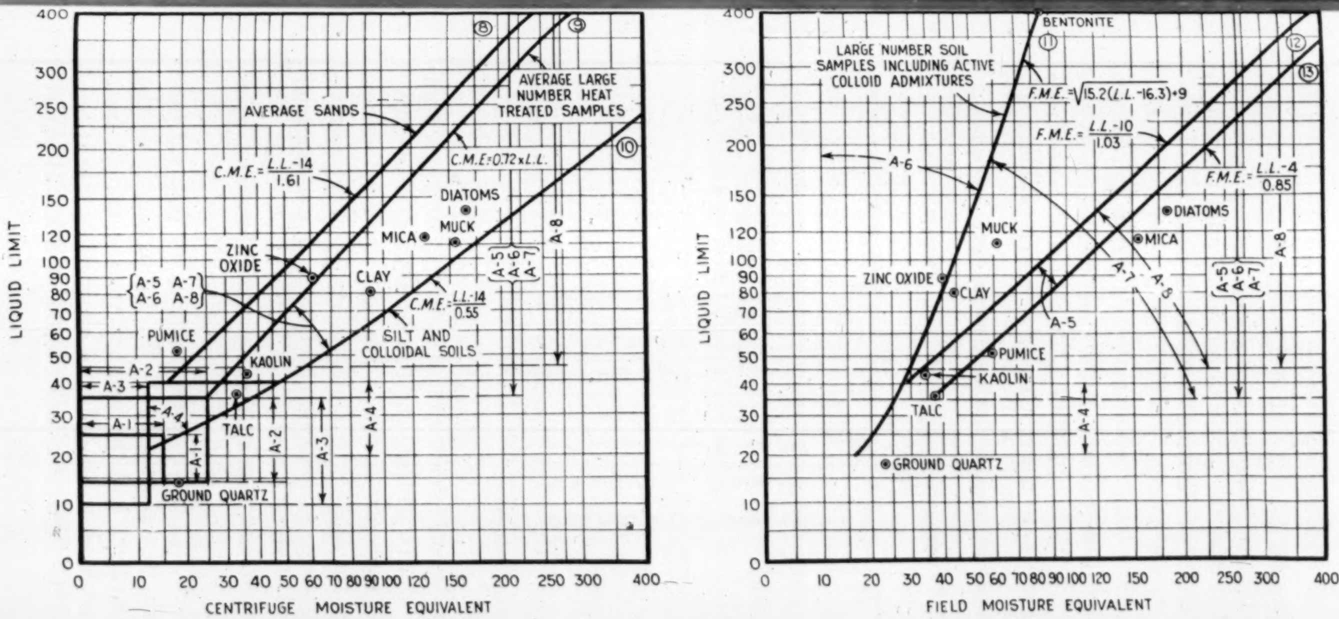

ELASTICITY
COHESION
COMPRESSIBILITY
DETRIMENTAL CAPILLARITY
A-8
PEATS AND MUCKS
A-7
MICACEOUS, DIATOMACEOUS AND FLOCCULATED CLAYS
A-6
COHESIVE CLAYS
DISPERSED STATE
A-4
FRIABLE CLAYS
COHESIONLESS SILTS
A-5
MICACEOUS AND DIATOMACEOUS SILTS AND SANDS
A-2
MATERIALS STABLE AT TIMES
A-3
SAND
INTERNAL FRICTION
A-1
GOOD GRADED MATERIALS

Fig. 23.—Diagram Illustrating Characteristics of Uniform Subgrade Groups.


LIQUID LIMIT
400
300
250
200
150
120
100
90
80
70
60
50
40
30
20
10
0
SHRINKAGE LIMIT
0
10
20
30
40
50
60
70
80
90
100
120
150
200
250
300
400
BENTONITE
⑤
$S = 21 - 1.1\sqrt{L.L. - \frac{L.L.^2}{800}}$
SILT CLAY AND COLLOIDAL SOILS
A-7
A-6
A-4
CLAY
TALC
MUCK
ZINC OXIDE
KAOLIN
PUMICE
GROUND QUARTZ
A-4
A-1
A-1
⑥
$S = \frac{L.L. + 86}{4.1}$
MUCK PEATS AND KAOLINS
DIATOMS
⑦
$S = \frac{L.L. + 26}{1.24}$
DIATOMS AND MICA
MICA
A-5
A-8
A-5 A-6 A-7


LIQUID LIMIT
400
300
250
200
150
120
100
90
80
70
60
50
40
30
20
10
0
PLASTICITY LIMIT
0
10
20
30
40
50
60
70
80
90
100
120
150
200
250
300
400
①
MICA
PUMICE
TALC
A-4
A-1
A-2
SAND, SILT, MICA, PEAT OR DIATOMS DOMINATING
$P.I. = 0.25 \times L.L.$
A-5 A-8
DIATOMS
KAOLIN
GROUND QUARTZ
②
$P.I. = \frac{L.L. - 14}{1.60}$
ZINC OXIDE
MIXTURES OF CLAY COLLOIDS AND ELASTIC MATERIALS
③
A-7
MUCK
CLAY
$P.I. = \frac{L.L. - 14}{1.07}$
SOILS CONTAINING COLLOIDS FULLY ACTIVE
BENTONITE
④
A-8
A-5 A-6 A-7
A-4
A-3
A-2
A-1



(c) (d)

FIG. 24.—Subgrade Soil Identification Chart.

Different locations on the diagram represented different combinations of the five basic soil properties with the A-3 subgrade of maximum internal friction located in the center.

As the position shifts from the center towards the outer boundary, the internal friction decreases. Shifting along the circumference from the bottom to the top, the soil changes gradually from the compressible to the elastic type. Adding compressible materials gradually changes a group A-3 sand to first a non-plastic variety of the A-2 subgrade; second, a well-graded stable A-1 subgrade; third, a plastic variety of the A-2 subgrade; and lastly, to either a group A-4 high-capillary silt or a group A-6 plastic clay which shrinks considerably and has high cohesion. The master charts utilizing the test constants for identifying soils of the various groups are shown in Fig. 24.

Table VI.—Illustrating Manner in Which Results Are Reported.

Sample	Material	Physical Characteristics Material Passing No. 40 Sieve						Lineal Shrinkage, per cent	Mechanical Analysis						
										Particles Smaller Than 2.0 mm., per cent by weight					
		Liquid Limit	Plasticity Index	Shrinkage Limit	Shrinkage Ratio	Centrifuge Moisture Equivalent	Field Moisture Equivalent		Particles Larger than 2 mm.	Coarse Sand, 2.0 to 0.25 mm.	Fine Sand, 0.25 to 0.05 mm.	Silt, 0.05 to 0.005 mm.	Clay Smaller than 0.005 mm.	Colloids Smaller than 0.001 mm.	Passing No. 40 Sieve
No. 8178....	Bentonite..................	399	354	6	2.2	964[b]	86	29	0	0	2	12	86	74	100
No. 8173....	Diatoms..................	136	21	125	0.6	165	184	10	0	0	2	22	76	30	100
No. 8171....	Mica..................	116	0	165[a]	0.5	129	156	0	0	0	6	81	13	8	100
No. 8170....	Muck..................	112	73	33	1.4	155[b]	61	11	0	0	8	19	73	47	100
No. 8177....	Zinc oxide..................	89	54	31	1.9	61	40	4	0	0	0	2	98	89	100
No. 7588....	Clay soil..................	80	51	13	1.9	91[b]	44	15	0	0	1	21	78	39	100
No. 8174....	Pumice..................	52	0	57[a]	1.0	18	59	1	0	0	24	67	9	5	100
No. 8172....	Kaolin..................	43	15	37	1.3	36	34	0	0	0	0	36	64	29	100
No. 8175....	Talc..................	36	11	29	1.4	33	37	4	0	0	6	73	21	7	100
No. 8176....	Quartz flour..................	18	0	26[a]	1.6	14	23	0	0	1	35	51	13	6	100

[a] Computed from shrinkage ratios and specific gravity.

[b] Waterlogged.

Table VI illustrates the manner in which the results of the routine subgrade tests are reported. Complete discussion of the essential features, together with the significance of the physical soil tests, has been published in *Public Roads*, Vol. 12, Nos. 4 and 5, June and July, 1931.

Complete procedures for preparing the samples for test, for performing the liquid limit, the plastic limit, the shrinkage limit, the centrifuge moisture equivalent and the field moisture equivalent tests and for making the mechanical analysis, as adopted by the Committee on Materials of the American Association of State Highway Officials for proposed tentative standards, have been referred to the Society's Committee D-4 on Road and Paving Materials.

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(15) Henry Aaron, "A Study of Hydraulic Fill Settlement," *Public Roads*, Vol. 15, No. 1, March, 1934. See Table 2, p. 5, for comparison of liquid limits and moisture contents of undisturbed samples of muck.

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DISCUSSION

MR. J. W. KUSHING[1] (*presented in written form*).—An analysis of specifications for mineral aggregate wearing courses, using clay as a binder, indicates that they do not give very much attention to fundamental principles governing the stability of such mixtures. Early research work in determining a proper mixture and proper grading of material for such surfaces, using calcium chloride as a bonding aid, led workers in this field to recognize the merits of tests which were being developed in the analysis and classification of subgrade materials.

Test methods were studied to determine their usefulness in connection with stabilization of wearing courses in mineral aggregate roads. Generally speaking, stabilization refers to methods of increasing the stability of this type of material by the balancing of soil properties. Our investigations in Michigan have shown that the soil property most frequently lacking in our gravel roads is cohesion, and that this condition can be corrected by the proper addition and mixing of clay. In this work the soil test value that serves as a measure of cohesion, namely, the plasticity index, has proved almost indispensable. Since this test measures the ability of the combined fine materials to retain moisture without destroying their cohesion, it is useful in making certain that the filler material between the aggregates in wearing courses has sufficient stability to hold the coarser materials in place. Other tests such as for shrinkage limit and field moisture have shown little significance in the study of stabilized wearing courses.

The mechanical analysis as it has been extended by the hydrometer method to measure the particle size of the soil fines seems to be of primary importance in determining the properties of the finished stabilized mixtures.

The following procedure is followed in specifying the proportioning of materials to be used in stabilization:

1. The gravel or sand to be stabilized usually contains little or no cohesive material. Samples are screened on the $\frac{3}{4}$-in., No. 4, No. 10, and No. 40 sieves. If the material passing the No. 40 sieve usually designated "soil fines," contains cohesive material, the plasticity index is determined.

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[1] Research and Testing Engineer, Michigan State Highway Dept., Lansing, Mich.

2. The clays available for stabilization are dried, pulverized, screened through the No. 40 sieve, and the plasticity index of the soil fines is measured.

3. Mixes of the soil fines from the gravel and from the most suitable clay are combined to determine the amount of the latter required to give a plasticity index of 9 to 11 to the finished mixture.

The following table is a typical example of a mechanical analysis and tests on a clay and a road surface before and after stabilization:

	Mechanical Analysis, per cent by weight								
	Retained on 1-in. Screen	Passing 1-in. Screen. Retained on ½-in. Screen	Passing ½-in. Screen, Retained on No. 10 Sieve	Coarse Sand Passing No. 10 Screen, Retained on No. 40 Sieve	Fine Sand Passing No. 40 Screen, Retained on No. 270 Sieve	Silt	Clay	Liquid Limit	Plasticity Index
Before stabilization	0	5	31	26	25	7	6	15	0
After stabilization	0	8	34	20	20	8	10	19	7
Clay.............	0	0	4	4	26	20	46	41	23

Michigan State Highway Department specifications for a finished mixture are as follows:

> The finished mixture shall consist of coarse aggregate, fine aggregate and natural soil binder, supplied separately or in combination, to conform to the following composition limits, by weight:
>
> | | |
> |---|---|
> | Passing ¾-in. circular opening screen, per cent.............. | 100 |
> | Passing No. 10 sieve, per cent........................... | 40 to 60 |
> | Passing No. 40 sieve, per cent........................... | 20 to 50 |
> | Passing No. 270 sieve, per cent.......................... | 10 to 25 |
>
> The fraction passing the No. 270 sieve shall not be more than two-thirds of the fraction passing the No. 40 sieve. The fraction passing the No. 40 sieve shall have a plasticity index between 6 and 14 as determined by the physical test methods of the U. S. Bureau of Public Roads. (Described in *Public Roads,* Vol. 12, No. 8, October, 1931, p. 203.)
>
> The proportions of coarse aggregate, fine aggregate, and natural soil binder, required to give the finished mixture described above, will be determined by laboratory tests on the materials to be used.

In conclusion, it may be stated that provided construction methods outlined for this type of work are followed, satisfactory results will be secured when the following requirements are met:

1. The plasticity index of the surface materials must be over 5, and the wearing course must contain 3 to 4 tons per mile of calcium chloride to hold moisture bond.

2. (*a*) When the plasticity indexes of wearing surfaces are 5 or more and carry a loose cover, clay binder should be added to consolidate loose material to the road surface.

(*b*) When the plasticity indexes of wearing surfaces are less than 5, sufficient clay binder must be added to stabilize to a depth of 3 in.

3. The plasticity index of the finished mixture should be over 10 in order that water penetration be reduced to a minimum, thereby obtaining maximum stability in wearing surface.

MR. G. A. RAHN[1] (*presented in written form*).—We have made practical applications of the soil test procedure, outlined by Mr. Hogentogler, in the improvement of soil roads, in developing the soils through the medium of admixtures from the lower into the higher classifications, or in test parlance from the A-4 or A-6 into the A-1 or A-2 groups. In this work exclusive attention was focused on the soil mortars, or that portion passing the No. 10 sieve, ignoring the portion above that size and eliminating everything above the 1½-in. size. Briefly the work can be divided into the following operations: (1) surveying, (2) testing and determining mix, and (3) construction.

Surveying.—As this type of work is of such a nature as not to justify the refinements of a subsoil survey, typified by the soil profile, a superficial survey was decided upon. This consisted in examining the soil in the surface of the road and in the banks adjacent to the road. A sample was taken of each formation encountered, and its extent determined. The sampling operation consisted of digging a hole to a depth of 12 in. on the center line or quarter point of the road, and lifting an 8 to 10-lb. sample representative of this 12-in. depth.

At the time of the survey an investigation relative to the availability of various admixtures (sand, stone screenings, slag screenings, etc.) both local and commercial was also carried on.

Testing and Determining Mix.—The soil samples, together with samples of available admixtures, were brought into the laboratory for test. The soils were subjected to the various tests enumerated by Mr. Hogentogler and placed in their proper groups. The admixtures were subjected to the hydrometer analysis.

With this information available, attention was turned to the gradations on both the soils and the admixtures as determined by the hydrometer analysis, and a combination of soil and admixtures developed which will place it in the gradation range of the A-1 group, if economically possible, or in the A-2 group closely approaching the A-1. The poorest soil in the section under study was next combined with the calculated percentage of admixture, and the physical constants and gradation determined. If this check proved satisfactory it was assumed that the mixture was correct.

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[1] Research Engineer, Pennsylvania Department of Highways, Harrisburg, Pa.

Construction.—During construction, control tests were carried out to make sure that the mixture intended was being secured. Simple field washout tests were made on samples taken at intervals whereby the amount of coarse and fine sand was determined.

The accompanying Table I illustrates the method of control practiced in two construction projects, and includes both the mechanical gradation and physical constants. Under each section the average soil gradation, the computed mix, the laboratory mix, and the field mix are given. In project B the samples were taken at intervals of 100 ft. throughout the project. Field mix samples were taken in the same location after mixing operation had been concluded. Attention is called to the close check possible between the computed mix and the field mix. Also the check between the constants represented by the laboratory mix and the field mix. This proves conclusively that this method of testing can be applied to control as well as to preliminary classification.

TABLE I.—SOIL-ROAD CONSTRUCTION CONTROL.

	Coarse Sand			Fine Sand			Silt			Clay			Liquid Limit	Plasticity Index	Shrinkage Limit	Shrinkage Ratio	Centrifuge Moisture Equivalent	Field Moisture Equivalent
	2.0 mm.	0.84 mm.	0.42 mm.	0.25 mm.	0.105 mm.	0.074 mm.	0.05 mm.	0.02 mm.	0.008 mm.	0.005 mm.	0.002 mm.	Colloids, 0.001 mm.						
PROJECT A:																		
Admixture	100.0	78.3	56.4	47.9	38.8	34.0	28.5	17.5	10.0	7.0	3.0	1.0	....	....	....	....	....	....
Laboratory;																		
1 soil: 1 admixture	100.0	88.4	74.0	67.7	59.8	54.8	48.5	35.0	24.0	19.0	12.0	7.5	20.4	5.3	14.0	1.98	18.6	16.6
Field (composite);																		
1 soil: 1 admixture	100.0	92.4	76.2	68.0	55.7	48.3	41.5	32.0	18.5	14.5	7.0	5.5	20.7	3.3	16.0	1.92	17.7	16.6
PROJECT B:																		
Admixture	100.0	79.8	28.2	7.9	3.6	2.6	....	....	....	....	....	....	....	....	....	....	....	....
Section No. 1:																		
Soil, average	100.0	98.8	95.8	88.0	72.0	65.3	59.4	46.7	32.9	25.9	15.0	8.6	....	....	....	....	....	....
Computed mix 1:1	100.0	96.5	81.8	61.7	43.0	38.5	34.6	26.8	18.4	14.4	8.7	5.3	....	....	....	....	....	....
Laboratory mix 1:1	100.0	93.6	73.8	58.4	48.0	45.9	45.5	38.5	27.0	20.0	10.0	6.0	23.8	7.1	14.6	1.89	22.0	18.4
Field mix 1:1	100.0	94.4	76.1	57.6	40.8	36.3	33.8	27.8	20.4	15.7	9.3	5.9	22.7	5.3	15.3	1.88	16.9	17.4
Section No. 2:																		
Soil, average	100.0	99.2	97.9	94.5	87.8	85.4	80.5	69.0	52.0	42.5	26.0	16.0	48.0	24.6	13.6	1.92	29.4	26.0
Computed mix 2:1	100.0	95.8	78.4	55.2	39.3	36.3	33.4	27.7	20.0	16.2	10.3	6.7	....	....	....	....	....	....
Laboratory mix 2:1	100.0	92.6	67.8	48.6	36.7	33.8	32.0	28.0	23.0	21.0	11.0	7.0	22.0	3.4	14.7	1.89	19.6	17.1
Field mix 2:1	100.0	95.2	78.1	59.3	41.5	37.0	35.0	29.5	23.0	18.5	10.5	7.0	24.2	8.1	14.0	1.89	15.9	17.6
Section No. 3:																		
Soil, average	100.0	99.3	97.9	95.9	90.4	86.5	83.2	71.3	54.8	45.2	29.1	19.9	....	....	....	....	....	....
Computed mix 2:1	100.0	95.8	78.4	55.6	39.9	36.6	34.3	28.4	20.9	17.1	11.3	8.0	....	....	....	....	....	....
Laboratory mix 2:1	100.0	91.4	66.5	50.2	41.2	38.8	38.5	34.5	25.0	20.0	13.5	10.0	26.0	9.4	13.7	1.90	18.9	17.9
Field mix 2:1	100.0	95.3	77.7	58.4	41.7	36.8	33.9	28.3	21.3	16.5	9.1	4.8	22.4	4.9	15.1	1.87	15.1	16.8
Section No. 4:																		
Soil, average	100.0	99.4	98.5	97.0	94.2	92.8	91.5	83.5	63.7	53.0	33.7	22.0	....	....	....	....	....	....
Computed mix 3:1	100.0	95.4	76.1	50.9	34.5	32.0	30.2	26.1	18.9	15.5	10.3	7.0	....	....	....	....	....	....
Laboratory mix 3:1	100.0	89.8	59.8	40.3	30.1	27.4	27.0	24.0	18.0	15.0	10.0	5.0	20.2	2.7	15.4	1.85	18.6	17.0
Field mix 3:1	100.0	94.0	73.8	53.2	37.9	33.7	31.8	27.0	20.5	17.0	11.5	7.0	23.0	5.8	14.2	1.91	17.3	16.6

MR. W. S. HOUSEL.[1]—There are two problems in highway construction to which the development in applied soil mechanics and methods of measuring soil resistance have application of particular importance:

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[1] Research Consultant, Research and Testing Division, Michigan State Highway Dept., University of Michigan, Ann Arbor, Mich.

namely, (1) the determination of the physical properties and behavior of subgrades, and (2) the measurement of the so-called stability of materials used in the building of road surfaces, particularly low-cost road construction which makes use of numerous combinations of granular materials with the addition of various types of binding material.

In connection with the resistance of road surfaces to failure under surface loads and in connection with the supporting capacity of subgrades, the "bearing capacity" or ability to support loads without permanent or progressive deformation is a physical property of primary importance. Although it may be related to a number of secondary properties, bearing capacity may be subject to direct measurement which may result in the elimination of a number of complications with much practical benefit.

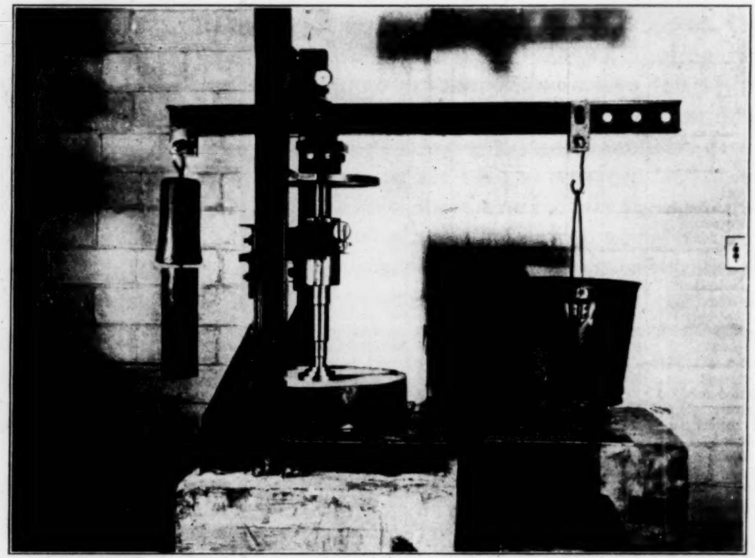

FIG. 1.—Machine Designed to Measure Capacity of Various Materials to Sustain Surface Loads.

Just how much application bearing capacity tests have to subgrade behavior seems to be a matter of some question since the bearing values of almost any of the soils encountered in highway construction seem to be sufficient to carry the low intensities of load to which they are subjected. But there may be some value in connection with subgrades in physical tests of this nature, particularly in cohesive soils with low resistance which serve as a foundation for flexible surfaces which transmit rather high intensities of load to the subgrade. However, direct physical tests of supporting capacity have their major application in the so-called stability of actual road surface material.

The Michigan State Highway Department is engaged at the present time in developing tests of this nature. The first investigation on "The Stability of Granular Materials," is being conducted by D. S. Berry at the Michigan State Highway Laboratory, the object of which is to develop adequate testing apparatus and to ascertain some of the fundamental elements of stability which do not seem to be adequately defined.

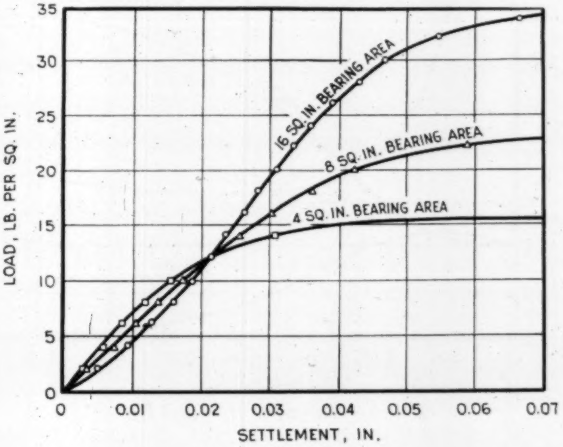

FIG. 2.—Load-Settlement Diagrams. Ottawa sand compacted by vibration.

The accompanying Fig. 1 is a photograph of a machine designed to measure the capacity of various materials to sustain surface loads. It consists primarily of a balance beam which furnishes a means of loading various sized bearing areas and has a total loading capacity of approximately 5000 lb. Settlement measurements may be made to an accuracy of 0.001 in. While the machine is not readily portable it has been found very convenient for a laboratory set-up and occupies very little space. Of more importance is the fact that the design and care in manufacture supplies a much higher accuracy than has usually been considered necessary in testing materials of this nature.

In its early stages the investigation has consisted of conducting bearing capacity tests on Ottawa sand and some tests have been conducted using several sizes of lead shot, the object being to determine the manner in which soil develops resistance to displacement as influenced by such factors as particle size and shape, degree of compaction, and other conditions which are characteristic of such mixtures. The accompanying Fig. 2 is an example of the typical results obtained from such tests using various sizes of rigid bearing areas which have been loaded to progressive failure. It should be noted that the bearing capacity at any given amount of settlement varies through a rather wide range for the different sizes of bearing area. For small amounts of vertical settlement the bearing capacity is greater for the smaller areas while in the upper range of settlement the bearing capacity is greater for the larger areas. The variation in bearing capacity for these granular materials follows the same general principles which have been determined by a large number of bearing capacity tests on cohesive materials.[1]

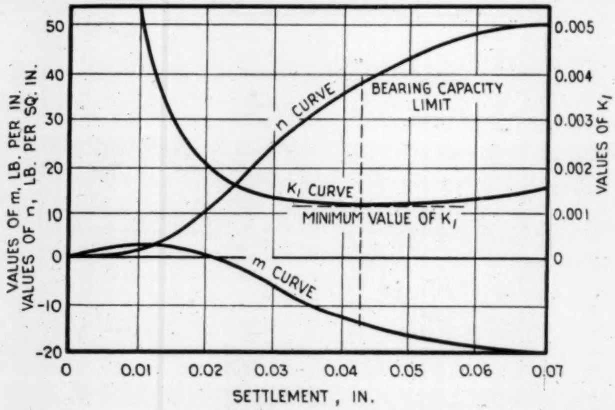

Fig. 3.—Values of m, n and K_1.

An analysis of the load-settlement diagrams at given amounts of settlement shows that the bearing capacity of such granular material may be expressed by a straight-line equation in which the bearing capacity is equal to the summation of two factors of soil resistance, which in previous investigations have been designated as perimeter shear and developed pressure. It appears quite obvious that if there is a variation in bearing capacity with the size of the area there must be some factor of soil resistance which is dependent upon dimensional properties of bearing area which are fundamentally the result of special boundary conditions.

If there were no special boundary effects to be considered the bearing capacity of all sizes of area should be the same. The conclusions drawn from quite a number of investigations made in this country and in Europe have been that the bearing capacity of granular materials is not affected by the size of the bearing area. At the same time it has been recognized that cohesive materials such as clay do exhibit properties which lead to such a variation in bearing capacity. The results shown in Fig. 2 represent, so far as I am aware, the first data from tests on granular materials which have been made with sufficient accuracy to expose the actual behavior of granular material for very small amounts of settlement.

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[1] W. S. Housel, "Bearing Power of Clay Is Determinable," *Engineering News-Record*, **February** 23, 1933, p. 244.

The straight-line equation which has been found to reproduce the various load-settlement curves accurately is given in Eqs. 1 and 2:

$$W = Ap = mP + nA \qquad (1)$$

$$p = \frac{W}{A} = \frac{mP}{A} + n. \qquad (2)$$

where W = total load,

m = perimeter shear in pounds per lineal inch,

P = perimeter in inches,

n = developed pressure in pounds per square inch,

A = area in square inches, and

p = bearing capacity in pounds per square inch.

The two factors of soil resistance are perimeter shear, m, and developed pressure, n. The developed pressure is due to the resistance of the material to volume change or consolidation, and the perimeter shear, m, is the result of resistance to displacement of the soil particles on the boundary planes. The total load on the bearing area is equal to the shearing resistance on the boundary multiplied by the perimeter plus the developed pressure multiplied by the area. In order to express the supporting capacity in terms of bearing capacity in pounds per square inch it is necessary to divide Eq. 1 by the area. In Eq. 2 the perimeter area ratio, $\frac{P}{A}$, expresses the dimensional properties of the bearing area in the same way that the hydraulic radius expresses the dimensional properties of a channel.

In the accompanying Fig. 3, the values of m and n, as determined from the analysis of load settlement diagrams, are shown for the entire range of settlement covered by the tests shown in Fig. 2. In the lower range of settlement the boundary effect is a positive factor in the bearing capacity while in the upper range of settlement the boundary becomes a source of weakness, indicated by negative values of the perimeter shear. This phenomenon may be accounted for by the fact that in the higher range of loads there is not sufficient lateral resistance to prevent displacement of the particles from beneath the edge of the bearing area.

The developed pressure, n, is not a large portion of the capacity for the smaller settlements as some consolidation of the material must take place in order to develop the resistance to compression. The maximum bearing capacity is determined by the ratio of settlement divided by the developed pressure, a function analogous to the coefficient of compressibility. This ratio was defined as the coefficient of settlement, K_1, and its critical value, which determines the bearing-capacity limit, has been fully discussed in connection with previous investigations.[1]

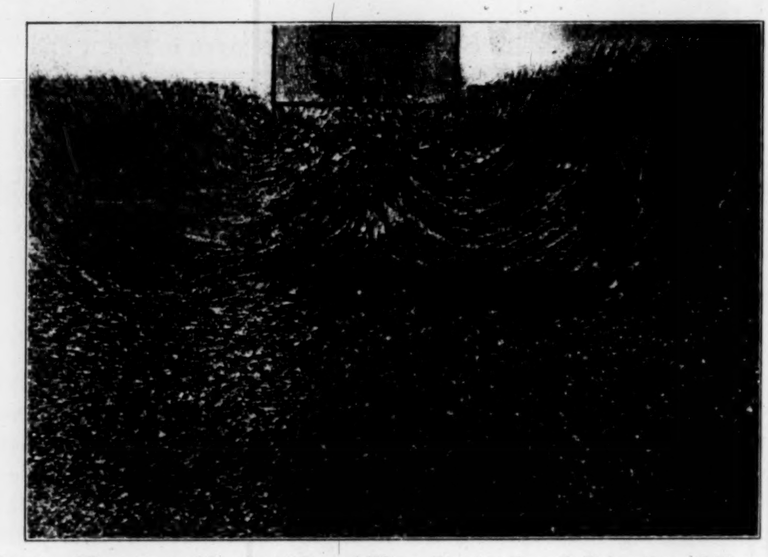

FIG. 4.—Movement of Sand and Gravel Where Bearing Area Is Being Forced Through the Material.

In a discussion of the bearing capacity of road surfaces the boundary effect or perimeter shear is a factor of considerable significance. In the first place, it is an indicator of the pressure distribution on the bearing area itself. If there were no boundary effect, the pressure on the bearing area would necessarily be uniform. However, when the boundary effect is positive it indicates that the intensity of pressure near the edge of the bearing area is greater than at the center, and conversely, when it is negative the pressure at the edge of the bearing area is less than at the center. In any investigation which has as its objective a determination of the manner in which pressure is distributed through road surfaces the variation in pressure on the bearing area itself is of primary importance and must be fully understood if any stress analysis in the material or distribution of load through the material is to be successful. When the investigation goes further into an analysis of the manner in which resistance is developed in order to determine the maximum and safe bearing capacities of the road surface, the factors of soil resistance which have been outlined are the most essential elements in this study.

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[1] W. S. Housel, "Bearing Power of Clay Is Determinable," *Engineering News-Record*, February 23, 1933, p. 244.

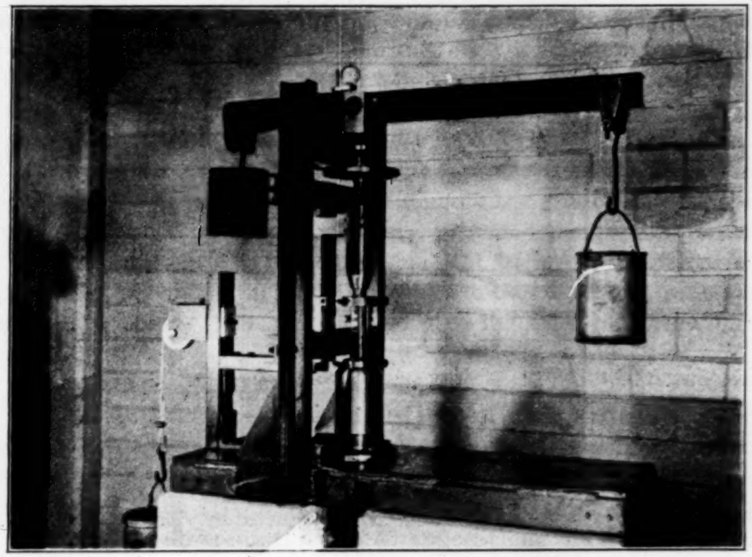

FIG. 5.—Testing Apparatus for Measuring the Shearing Resistance of Soils.

One of the most important conclusions which appears to be supported by the results of both tests on cohesive and granular is that the bearing capacity limit or ultimate supporting capacity is controlled primarily by the resistance of the material to lateral displacement, which is fundamentally shearing resistance. It is also important to realize that progressive settlement which results when the bearing capacity limit is exceeded is in all respects in agreement with the phenomenon known as plastic flow. The accompanying Fig. 4 is a photograph made by taking a time exposure of the movement of particles of sand and gravel while a bearing area is being forced through the material and indicates the manner in which displacement of the material takes place.

There are two theories of soil resistance under consideration at the present time which are calculated to describe the behavior of a body of material under superimposed loads. These theories may be designated as the "Consolidation Theory" and the "Theory of Plastic Flow." The consolidation theory pictures the settlement under load as entirely due to volume change or compression of the soil particles sometimes accompanied by the squeezing out of water which fills the voids in the soil. The conclusions drawn from this theory are that soil has no definite bearing capacity but that progressive settlement under any superimposed load will continue until the material reaches its maximum state of consolidation. The theory of plastic flow pictures settlement under any superimposed load as a combination of consolidation and displacement and leads to the conclusion that soil does have a definite bearing capacity and is capable of sustaining loads within the bearing capacity limit without progressive settlement after the initial consolidation has taken place. The most important consideration in this latter theory of bearing capacity is that the shearing resistance of the material, which might also be referred to as the plastic limit, sets a definite limit to the supporting capacity which has been defined as the bearing-capacity limit.

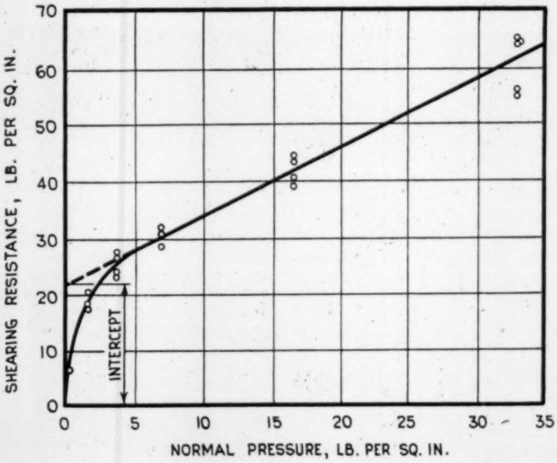

FIG. 6.—Shear Test. Fine sand compacted by vibration.

In addition to the bearing capacity tests being conducted, tests to measure the shearing resistance of soils of various types have been devised. The testing apparatus is shown in Fig. 5. The apparatus provides means for applying vertical load to a soil sample held in a cylindrical mold. Means are provided for measuring the transverse shearing force necessary to pull out a section of the cylinder under varying intensities of normal force. This test is modeled after similar tests made by Bell in 1914 and reported in the *Proceedings* of the Westminster Institute of Civil Engineers. The preliminary results of tests run on Ottawa sand and some other granular materials indicate several interesting facts. First, a body of granular material has a definite shearing resistance, shown as the intercept in Fig. 6, due to interlocking of the grains, which to some degree is independent of the normal pressures. Shearing failure is abrupt and is accompanied by a measurable volume expansion in agreement with principles that have previously been outlined by Dr. K. Terzaghi. There is an increase in the shearing resistance with increased normal pressure in addition to the initial resistance due to interlocking, but this increase cannot always be expressed by a coefficient of friction with a constant value. In Fig. 6 is shown some typical results of the measurement of shearing resistance of fine sand under the conditions described. From these results it is apparent, that, provided the normal pressure on the shearing surface is merely sufficient to hold the grains in a compact arrangement the body of sand exhibits a definite resistance due to interlocking, which is analogous to the cohesion encountered in clay.

The tests described are merely the first steps in what is intended to be a comprehensive study of resistance of some materials used in highway construction. The method of approach used in these investigations is somewhat different from those outlined by Mr. Hogentogler, and I have attempted merely to present as much as possible of a somewhat different method of approaching these problems of mutual interest. It is our hope that the application of these methods to problems in highway materials will yield some valuable results.

MR. C. A. HOGENTOGLER.[1]—In closing, I wish to stress the importance of the work described in Mr. Housel's discussion. Our paper called attention to four different kinds of tests but attempted to present only a picture of what the so-called "simplified physical tests" can disclose, because the procedures for performing these tests have become fairly well established. Investigations along the line suggested by Mr. Housel are also considered in our soil stabilization work.

In the determination of grain size we find that the type of deflocculent used in the preparation of the sample is of utmost importance. Investigations reported in *Public Roads*, Vol. 14, No. 6,

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[1] Senior Highway Engineer, U. S. Bureau of Public Roads, Washington, D. C.

August, 1933, p. 95, Table 2, illustrate this. Here in one case the amount of clay is shown to be 67 per cent, 78 per cent or 87 per cent, depending on whether the dispersing agent is potassium hydroxide, sodium oxalate or sodium silicate. Also, the colloidal fraction (particles smaller than 0.001 mm.) may equal 0 per cent, 1 per cent, or 29 per cent, depending upon whether the dispersing agent is potassium hydroxide, sodium carbonate, or sodium silicate.

This work all points to the general conclusion that results furnished by mechanical analysis depend entirely on the arbitrary procedure employed. Any variation in the procedure may cause a change in the apparent grading of the soils.

Finally, since there has been so much said about recent developments in soil tests and research, I should like to point out that progress in this field has concerned developments of methods and employment of apparatus more than change in fundamental conceptions. As a matter of fact, Daniel E. Moran reports making consolidation tests on undisturbed samples of clay and other foundation materials as early as 1910. The conception of silt consisting of bulky grains and clay of scale-like particles as well as that of shrinkage being caused by surface tension when water recedes in the soil capillaries was published by George E. Ladd in 1898,[1] and in 1892 Milton Whitney[2] explained in detail the effect of surface tension for contracting, expanding, and supplying cohesion in soils; suggested a formula utilizing the relationship between surface tension, diameter of capillaries and height of capillary rise; and described a method for determining the moisture content by means of the changing electrical resistance between two plates buried permanently in the soil in place. These pioneers should receive full credit for their contributions which helped to make the present progress possible.

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[1] G. E. Ladd, "Phenomena Resulting from the Surface Tension of Water," *American Geologist*, p. 267 (1898).

[2] U. S. Department of Agriculture, Weather Bureau *Bulletin No. 4* (1892).

THE IMPORTANCE AND THE DETERMINATION OF PARTICLE SIZES OF HYDRAULIC-FILL DAM MATERIALS[1]

By H. H. Hatch[2]

As the height of earth dams has increased, the importance of minute technical study of the material, careful investigation of the strength and proper control of the structure during construction has become more evident.

The research at Cobble Mountain, both in the laboratory and in the field, was started with the full advantage of all the practical and theoretical knowledge available in connection with the control of hydraulic-fill dam construction. No method dealing with the problem on hand developed elsewhere was either adopted or discarded until after it was given a trial at Cobble Mountain. Perhaps for the first time in the history of engineering there has been a research of this type carried on side by side with actual construction.

The Importance of Particle Sizes

It was discovered that the particle size or the gradation is one of the most important investigations for the suitability of a material for a hydraulic-fill dam.

The function of the core section of a hydraulic-fill dam (Fig. 1) is to stop the passage of water through it. All earth dams leak more or less, and the seepage through a hydraulic-fill dam will depend almost entirely on the particle sizes of the core material. A core of finer particle sizes will leak less than one with larger core materials. But the smaller the particle size of the core material, the longer will it take to settle, drain, and consolidate, and *vice versa*. A core almost water-tight can be obtained with the required fineness of particle size, but this in turn will make the structure, unless with unnecessarily large section, rather unstable because of perhaps indefinite delay in settlement, drainage, and consolidation. Both of the required results, that is, safety and water-tightness, cannot be obtained at the same time. Therefore a compromise should be made so that a dam sufficiently water-tight is obtained with the required amount of safety for the structure. This means that the core material must have

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[1] This paper was presented in connection with a Symposium on Industrial Applications of Particle Size Measurement, sponsored by the Committee on Particle Size and Shape of the Society's Committee E-1 on Methods of Testing, held in conjunction with the fourth Regional Meeting of the Society, New York City, March 8, 1933.

[2] Engineer in Charge, Cobble Mountain Reservoir, Springfield Water Works, Westfield, Mass.

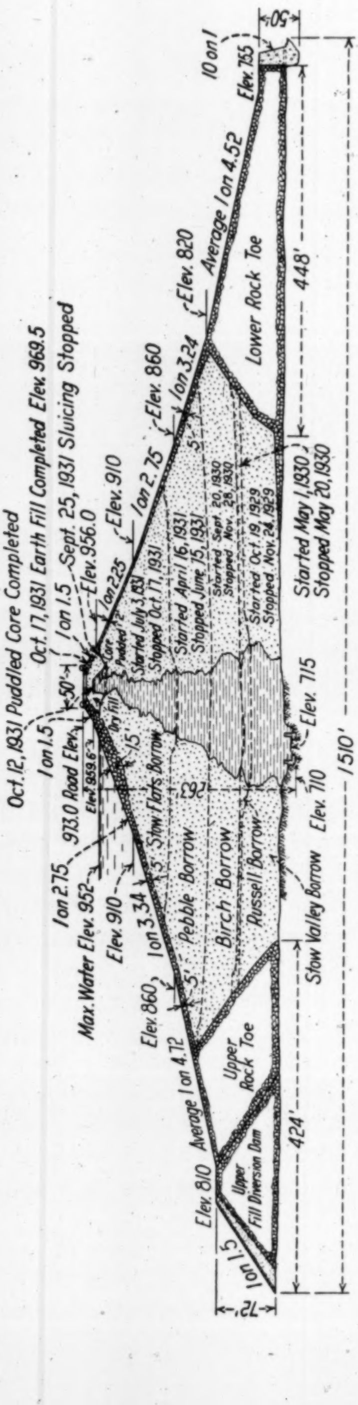
Oct. 12, 1931 Puddled Core Completed
Oct. 17, 1931 Earth Fill Completed Elev. 969.5
Sept. 25, 1931 Sluicing Stopped
Elev. 956.0
1 on 1.5
1 on 2.25
Elev. 910
1 on 2.75
Elev. 860
1 on 3.24
Elev. 820
Average 1 on 4.52
Elev. 755
10 on 1
50'
1 on 1.5
973.0 Road Elev.
Elev. 959.6
50'
1 on 2.75
Max. Water Elev. 952
Elev. 910
1 on 3.34
Elev. 860
Elev. 810
Average 1 on 4.72
1 on 1.5
72'
Core Puddled
Dry Fill
Started July 3, 1931
Stopped Oct. 17, 1931
Started April 16, 1931
Stopped June 15, 1931
Started Sept. 20, 1930
Stopped Nov. 28, 1930
Started Oct. 19, 1929
Stopped Nov. 24, 1929
Started May 1, 1930
Stopped May 20, 1930
Lower Rock Toe
Stow Flats Borrow
Pebble Borrow
Birch Borrow
Russell Borrow
Stow Valley Borrow
Upper Rock Toe
Upper Fill Diversion Dam
5'
15'
263'
Elev. 715
Elev. 710
448'
424'
1510'

FIG. 1.—Section of Cobble Mountain Dam.

particle sizes fine enough to prevent, within a certain degree, the seepage of water through it, and yet it must contain large enough particles in the material so that it will expedite the settling, the drainage, and the consolidation of the structure. Proper gradation is the key to the solution.

The function of the beaches of a hydraulic-fill dam is to support, during construction, the liquid or semiliquid core. The beaches, therefore, must not only be heavy enough to resist this pressure but also porous enough to permit the core to drain. The more uniform the particle size of the beach material, the higher will be the percentage of voids, but this results in less weight in the structure and lessens its stability both during and after construction. A better gradation or uniform variation in size will result in a smaller percentage of voids of the beach material, which results in a stronger structure. Hence, the importance of proper particle size control.

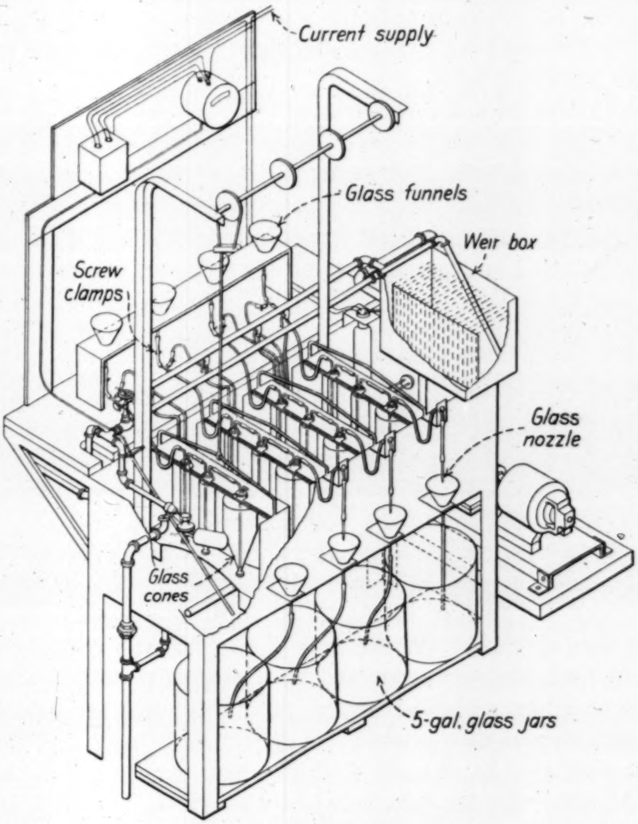

FIG. 2.—The Elutriator.

DETERMINATION OF PARTICLE SIZES

The mechanical sieve analysis with a No. 200 sieve as the finest sieve is limited to about 0.09 mm. The previous method of procedure for the accurate determination of grain sizes of material passing a No. 200 sieve by decantation or elutriation was either inadequate or very slow. The tests at Cobble Mountain were to serve the purpose of controlling the particle sizes of the core material during the construction and it became very necessary to develop a practical method that would take a reasonably short time to make the required tests with reliable results.

The Cobble Mountain elutriator (Fig. 2) consists of three glass cones 1, 2, and 4 in. in diameter, each provided with an inlet tube running to the bottom, and outlet tube running to the next cone. The first cone inlet tube has a connection to a glass funnel into which is placed the portion of the test material that has passed the No. 200 sieve. From this funnel the material is carried to the first, then the second, and then the third cone which has a connection to a glass nozzle discharging the overflow into a receptacle. With the arrangement of the inlet tubes a rising column of water of different velocity is created in each cone due to different cross-sections of the cones. When the sample material is washed into the cones, necessary connections are made for the constant and uniform flow of the water through the cones. As the material reaches the first cone, the larger particles settle and the smaller particles are carried over to the next. Similar action takes place in the other cones. The size of the particles settled in each cone has a definite relation to the cross-sectional area of that cone. The test is continued until the overflow discharge becomes almost clear. At the end of the run the particles at the bottom of each cone are recovered by a process of filtration through alundum crucibles under a vacuum, and the overflow is flocculated with chemicals and then filtered.

The elutriator has 4 units so that 4 tests can be run at the same time. All the units are submerged in water in a steel tank and provided with thermostatic control to keep the temperature constant, and a circulating pump to keep the flow uniform in all the cones. With a correspondingly larger tank, almost any number of elutriator units can be installed and as many tests run at the same time.

Principle of the Test:

The principle of the separation of particle sizes in the elutriator is based on Stokes' law. Stokes' law gives the constant velocity of falling bodies with a certain diameter through a liquid. In this case, however, the same law applies to bodies of the same diameter held in suspension by the upward flow of the same liquid. The following formula has been developed for the elutriator:

$$d = 0.0152 \left(\frac{f}{a}\right)^{\frac{1}{2}} \left(\frac{60}{t + 10}\right)^{\frac{1}{2}}$$

where d = average diameter in millimeters of a particle,
f = flow in cubic centimeters per minute,
a = area of the cone in square centimeters, and
t = temperature in degrees Fahrenheit.

To obtain the best results, it is necessary to have the flow and the temperature of the water uniform throughout the run of the test. In case there is a change in the temperature, the flow should be changed according to the formula in order to keep the value of *d* constant.

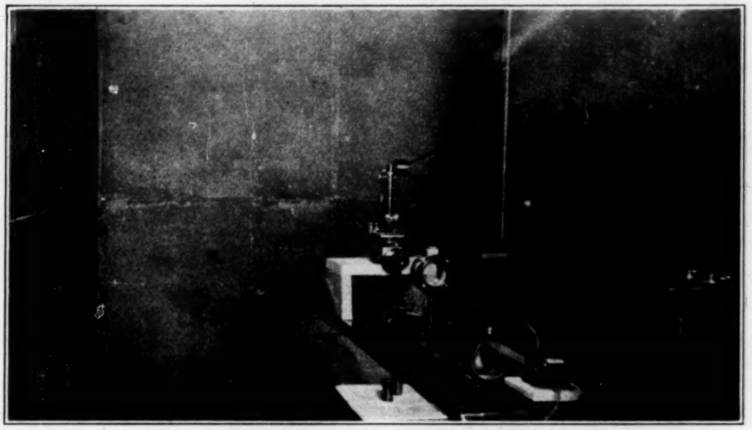

FIG. 3.—Microscopic Projection Apparatus.

It is seen from the elutriator formula that the particle size varies directly as the square root of the flow and inversely as the square root of the cone area. It should also be remembered that the duration of the test has an approximate inverse ratio to the flow. A flow of 48 cc. per minute at 77 F. will give a low separation of 0.01 mm. and the actual run of the test through the elutriator will take about four hours. Then with a flow of 12 cc. per minute a separation of 0.005 mm. can be obtained during about sixteen hours. According to the formula, no doubt within limits, during a shorter time larger separations can be obtained with higher flows. The elutriator with a flow of 48 cc. per minute at 77 F. gave separations of particle sizes 0.04, 0.02, and 0.01 mm. for 1, 2, and 4-in. diameter cones, respectively.

CHECKING BY MICROSCOPIC EXAMINATION

The results obtained by the elutriator are reliable, especially when they are checked now and then by microscopic examination. The microscopic measurements may involve assumptions which perhaps are not exact, and which may influence the results, and yet it is the most reliable method available for observation and checking purposes. The microscope is very useful for observing and studying the shape and other characteristics of particles.

The microscopic measurements of the separation of particle sizes of the No. 200 sieve and also of the three stages of the elutriator were made at Cobble Mountain in accordance with the method outlined by L. T. Work.[1] The microscopic apparatus (Fig. 3) consisted essentially of a projection lantern throwing light rays through the cooling cell, condensing lenses onto the substage mirror of the microscope. Then the light was reflected through a condenser and the slide containing the sample of soil into the objective lens of the microscope. By means of an adjustable prism near the ocular lens, the image of the particles is thrown on a painted plate-glass screen.

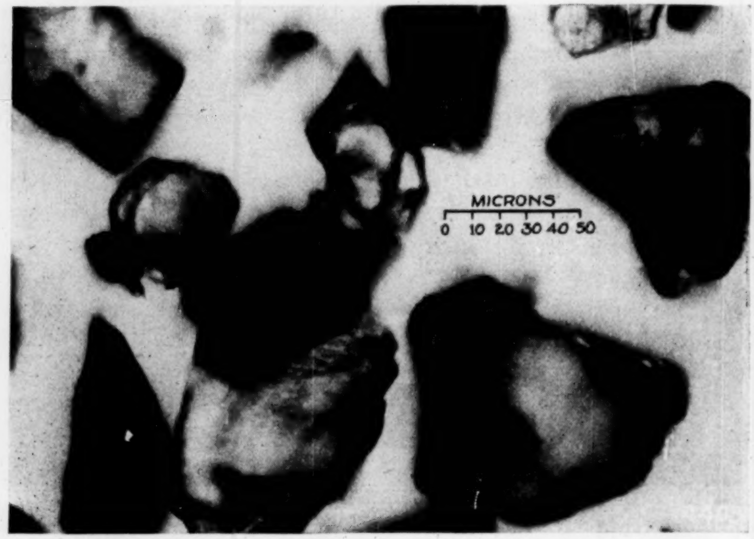

FIG. 4.—Elutriation, 1-in. Diameter Cone (Range 0.04 to 0.09 mm.).

By projecting a stage micrometer on the screen the magnification can be accurately measured. For any magnification on the screen a corresponding scale can be made to measure the dimensions of the particles directly (Fig. 4).

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[1] L. T. Work, "Methods of Particle Size Determination," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 780 (1928).

Slides were made of the samples to be tested and the image thrown on the screen. By moving the slide slightly, new fields are projected. The sample should be well dispersed and the particles evenly and distinctly spread over the field. They should not overlap one another. Only the good fields are considered and they are taken up separately. The width of all particles, except the mica, is measured and counted. The particle sizes are divided into several ranges and the frequency of each size is counted. About fifteen fields are measured, and their results summarized. The volume and weight of each size range are computed and also their ratio, per cent per micron, with respect to the entire sample. The ratios, per cent per micron, are plotted against size ranges in microns and this rectangular plotting is replaced by a graph in which the ratio, per cent per micron, is plotted against diameter sizes in microns. These curves overlap one another, due, perhaps, to shape of particle, indicating that a small amount of fines has been left with the coarser separation and similarly a small quantity of coarse material (especially flat particles) has been carried over to the next smaller size separation. Areas under the curve represent per cent by weight. By balancing the fines in a coarse separation with the coarse particles in the next fine separation, the division line between the elutriator stages can be obtained. As a rule, this division line is at or very near the intersection of the overlapping curves (Fig. 5).

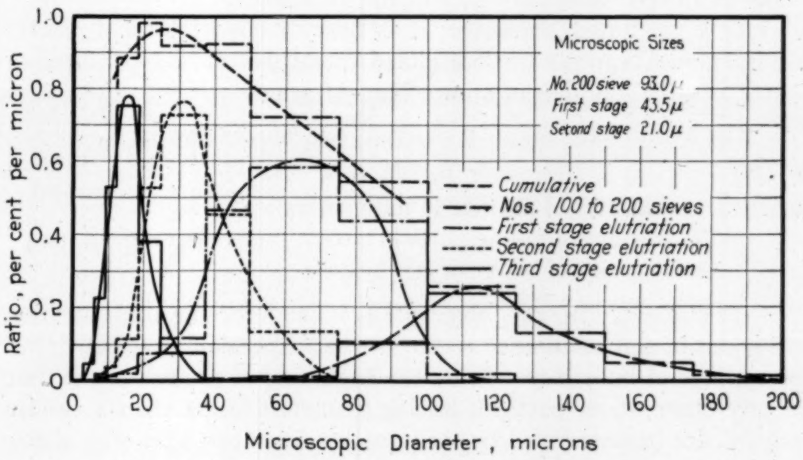

FIG. 5.—Microscopic Sizing of Elutriator Stages.

HYDROMETER METHOD

The most rapid method known to the author for determining the grain sizes below the No. 200 sieve size with reasonable accuracy is that using the hydrometer developed by J. G. Bouyoucos.[1] Within an hour an analysis can be made of a soil sample by this method. The apparatus required for the hydrometer method is very simple and its cost is almost negligible compared with that of the elutriator. This method has many advantages and it is being used in many places for determining the grain sizes of soil particles. The hydrometer is so constructed that the graduation on its stem at the water surface will indicate the number of grams of soil in suspension per liter of water at the center of buoyancy of the hydrometer.

As a precaution, however, it is best to check the hydrometer readings with various predetermined quantities of soil per liter of water, or with solutions of known concentration.

The hydrometer equation derived from Stokes' law is:

$$d = 0.1033\ (Vn)^{\frac{1}{2}}$$

where d = diameter of particle in millimeters,
V = velocity of falling particle in centimeters per second, and
n = viscosity of water in centipoises.

At different intervals observation is made of the concentration (in grams of soil per liter of water) of the suspension at the hydrometer center of buoyancy. From the beginning of the test to the time of any observation, particles having diameters larger than a certain critical size have traveled by, or particles finer than that critical size are still in suspension above the center of buoyancy. The traveled distance is taken from the water surface to the buoyancy center, but this is not constant for different concentrations of a suspension, nor is it necessarily the same for any two hydrometers. For each hydrometer a curve should be prepared showing the distance from the water surface to the buoyancy center for various readings on the stem of the hydrometer. With distances obtained from the curve and the time in seconds as observed, the velocity can be computed and the equation solved for d.

The hydrometer has been designed for a water temperature of 67 F. (19.44 C.). If water is used with another temperature it will be necessary to make a correction due to its change in density according to Fig. 6. The corrected hydrometer reading gives the weight of particles finer than those of *d*, for which the observation was made. This weight divided by the total weight of the original sample gives the percentage finer than *d*.

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[1] J. G. Bouyoucos, "Directions for Determining the Colloidal Material of Soils by the Hydrometer Method," *Science*, Vol. LXVI, No. 1696, July 1, 1927, pp. 16–17; and "Making Mechanical Analyses of Soils in Fifteen Minutes," *Soil Science*, Vol. XXV, No. 6, June, 1928, pp. 473–480.

With observations made at different time intervals, corresponding computations are completed and the gradation curve is plotted.

Some soils in their natural state contain elements which produce flocculation when the material is placed in pure water. In most cases this may be overcome by the use of a deflocculating agent (ammonia or sodium oxalate). For local materials which showed very slight, or no tendency to flocculate, a fixed amount (5 cc. per liter) was used satisfactorily.

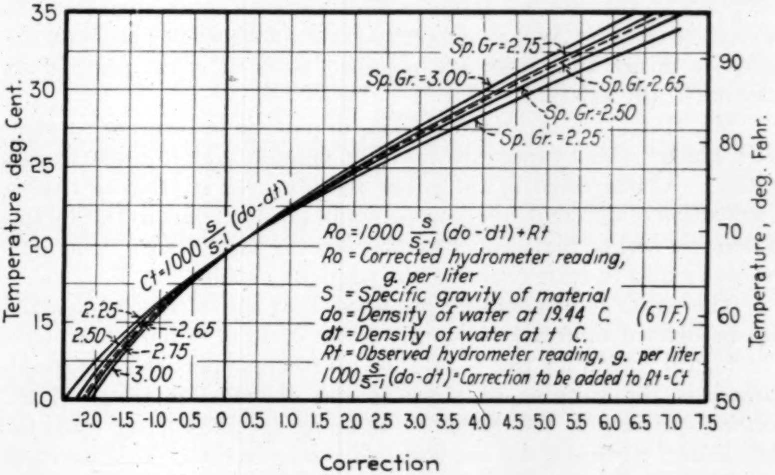

FIG. 6.—Temperature Correction Curves for Bouyoucos Hydrometer Readings.

The hydrometer method will give reasonably accurate results, at least for the gradation of hydraulic fill-dam materials, for readily dispersing and granular soils. Undoubtedly this method is very useful and of great help during periods when the results are necessary in the shortest time possible. For further details on the elutriator and hydrometer methods and the derivation of their formulas see "Tests for Hydraulic Fill Dams."[1]

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[1] H. H. Hatch, "Tests for Hydraulic-Fill Dams," *Proceedings*, Am. Soc. Civil Engrs., Vol. 58, No. 8, October, 1932, pp. 1301–1342.

DISCUSSION

MR. P. S. ROLLER[1] (*presented in written form*[2]).—I believe one of the first considerations in the accurate measurement of particle size is that of definition of the size of particle. While an arbitrary definition may be convenient, it may on the other hand confuse the issue to the extent that particle size as expressed is no longer an independent variable but includes effects due to other variables such as particle shape. As a result comparisons of different materials may be quite misleading, since the assumed basis of equal particle size may not really exist.

Mr. Hatch has expressed the size of particle in terms of its measured width. This procedure may be simplest but it leads to difficulties. As suggested in the paper the observed overlap of the size distribution of different fractions no doubt is due in part to this method of defining size of particle. Probably for the same reason, it is found necessary also to omit counting the particles of mica.

The writer has previously proposed[3] that the microscopic particle size be defined uniformly in terms of the ratio of surface to volume. The logical reason for this is that for microscopic particles the specific surface is the variable of physical and chemical interest. For a rectangular particle the definition based on specific surface leads to a size defined rigorously by the harmonic mean of the three dimensions.

The physical separation of the microscopic particles considered in Mr. Hatch's paper is based on Stokes' law. In this law the variable of size is equivalent to the ratio of surface to volume of the particle. It has been found that with the theoretical Stokes' law size the same, the superficial size may be quite different, depending on the shape of the grain. For example, particles of gypsum and slate appear larger than particles of anhydrite or portland cement, although the Stokes' law size is the same. However, when the particle size is interpreted in terms of the surface to volume ratio, the measured sizes then appear to be approximately equal to each other and to the Stokes' law sizes. This agreement is of importance, because the particle size defined in terms of specific surface now appears as a truly independent variable. A correct basis is thus provided for comparison of different materials with respect to the effect of particle shape, for example, keeping the particle size truly constant.

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[1] Associate Chemist, U. S. Bureau of Mines, Nonmetallic Minerals Experiment Station, New Brunswick, N. J.

[2] Published by permission of the Director, U. S. Bureau of Mines, Washington, D. C.

[3] P. S. Roller, "Separation and Size Distribution of Microscopic Particles—an Air Analyzer for Fine Powders," U. S. Bureau of Mines *Technical Paper 490* (1931).

Mr. Hatch's reference to the need of deflocculation in the hydrometer method can hardly be overestimated. In methods analogous to the hydrometer method, which involve single-stage sedimentation, checks against accurate direct analysis (by air)[1] could not be obtained without stringent deflocculation of small amounts of the material. Certain presumed deflocculating agents may be only partially effective. Owing to the apparent variation in soluble (gypsum, etc.) and in organic matter content of different core materials for hydraulic-fill dams, the question of deflocculation in single-stage sedimentation, and even in water elutriation, should be carefully considered for each material. It will be recalled that in elutriating kaolins Schurecht[2] found a wide difference in result depending on whether a deflocculant was or was not used in elutriation. Gross, Zimmerley and Probert[3] used gum arabic and a mechanical device for deflocculation.

Further information on the derivation of the hydrometer equation from Stokes' law would be highly desirable. Puri[4] has pointed out that the Bouyoucos hydrometer is not amenable to theoretical treatment because of the large bulk of liquid displaced. Only an indefinitely small volume of the displaced liquid will correspond to the "point in the column" that is required for mathematical correlation of particle size and density of the suspension.

MR. I. F. MORRISON[5] (*by letter*).—Apart from the purely research aspect, methods for the determination of particle size, where fine-grained material is concerned in construction, are of growing importance both from the point of view of preliminary investigations and control. The preliminary study should concern itself with a determination of the relative amounts of the coarse, fine, and very fine portions of the material available and with a study of the amount of fine material which will settle in a given time. The first studies can be made by any one of several methods recently developed which are easily available. The hydrometer method, as recently developed at the U. S. Bureau of Public Roads and by A. Casagrande and others, gives satisfactory results and a uniformity curve can be readily constructed for the material passing a No. 100 sieve. A study of this curve in comparison with those for hydraulic-fill cores already successfully constructed will reveal how much of this material must be wasted in order to obtain a suitable core. A suitable core material may have quite a wide range of particle size.

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[1] P. S. Roller, "Measurement of Particle Size with an Accurate Air Analyzer: The Fineness and Particle Size Distribution of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 607 (1932).

[2] H. G. Schurecht, "Elutriation Tests on American Kaolins," *Journal*, Am. Ceramic Soc., Vol. 3, p. 355 (1920).

[3] John Gross, S. R. Zimmerley and Alan Probert, "A Method for the Sizing of Ore by Elutriation," U. S. Bureau of Mines *Report of Investigations 2951* (1929).

[4] A. N. Puri, "A New Type of Hydrometer for the Mechanical Analysis of Soils," *Soil Science*, Vol. 33, p. 241 (1932).

[5] Professor of Applied Mechanics, University of Alberta, Edmonton, Alta., Canada.

The elutriator described by the author would seem quite suitable for the requirements of this preliminary study, with one exception. That is, only three points on the uniformity curve are determined by means of it. But, since the curve is of the ogee type, it is desirable to have not less than five points if a good comparison with the curves already obtained for hydraulic fill cores is to be made. The apparatus could be easily extended with little trouble so as to use 5 cones instead of 3.

For the purpose of such preliminary studies, it is of course necessary to use a deflocculating agent. However, as flocculation usually occurs in the natural process of construction, and since it would be inexpedient to attempt to prevent it in the actual construction, studies should be made of the rate of settlement preferably for the water which will actually be used during construction. There is at present no apparatus or established method for doing this but it can be accomplished by trial by mixing known quantities of the material in the water and draining off the turbid fluid after certain periods of time until the remaining material, when analyzed by the hydrometer will give a satisfactory uniformity curve for the core.

It will then be known at what rate the water from the pool must be drawn off in order to leave a suitable core. The core material, thus separated, should be further examined in order to determine its coefficient of permeability from which, along with its pressure-moisture curve, an estimate may be made of its rate of consolidation if thought necessary. Information of this sort is desirable because of its bearing on the rate at which the heavy toes, which usually exert some side pressure on the core, may be built up.

In the control of operations on the job, the chief requisite is the time element rather than great accuracy, although the limits of accuracy should be known. The control of the operation of the pool should be based on the amount of waste in the overflow water and the quality of the core produced. Both the overflow water and the core material should be sampled frequently. Since the core material, as compared with that in the overflow water, is relatively coarse, the elutriator described in the paper may be of value for the analysis of it provided the time required is not too long. On the other hand, no advantage over the simpler hydrometer method seems apparent for this purpose. Besides, the hydrometer method involves less, rather than more, time. As for the material in the overflow, all that seems necessary is to determine the weight of the material in suspension, which can be done immediately by the hydrometer and checked by evaporation and weighing.

MR. L. T. WORK.[1]—The laboratory tests of fineness at Cobble Mountain were devised for maximum accuracy with economical production. It was necessary to test a large group of samples each day in order to have a complete history of the case and to apply adequate correctives in the choice of soil to be used. The hydrometer was used for a great deal of this work although it possessed some elements of inaccuracy. It was checked with the elutriation and microscopic measurements. There was a marked correlation between the hydrometer values and the elutriator-microscope method so that the points which have been raised as to the shape of the soil particles were comparatively unimportant. The reference to Puri indicates an approach to perfection in the hydrometer method, but fundamentally it can never cure it for the hydrometer bulb must have a volume, and if it is flat in shape or even spherical, there is always the question of sedimentation on the glass. Further, the stem of the hydrometer must have a volume, and that action prevents the hydrometer from measuring density truly at a specific point.

In the problem at Cobble Mountain the spread of particle size was very great from millimeters down to microns and the entire span had to be measured. The application of the data was primarily to permeability, consolidation, etc., and since the shape factor of the particles was so uncertain and was difficult of exact measurement, they actually took the size measurements, as they could readily determine them, and correlated them with other laboratory and field tests necessary for the safety of the structure both during and after construction.

MR. H. H. HATCH[2] (*author's closure by letter*).—The author agrees with Mr. Roller that the shape of the particle is an important factor and perhaps it should be given weight in the particle size determination. No matter how desirable this adjustment may be from theoretical standpoint, it becomes almost impossible for practical purposes, especially in hydraulic fill construction where, with thousands of cubic yards of sluiced material going into the structure from various borrow pits, it is of utmost importance to make numerous tests quickly for the control of the construction. The questioned method of measuring particle sizes and also elements of inaccuracy of the hydrometer are comparatively unimportant, as pointed out by Mr. Work.

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[1] Assistant Professor of Chemical Engineering, Columbia University, New York City.

[2] Engineer in Charge, Cobble Mountain Reservoir, Springfield Water Works, Westfield, Mass.

The derivation of the hydrometer equation from Stokes' law, as requested by Mr. Roller, is as follows:

Stokes' law is expressed by the equation:

$$V = \frac{2\ (P_p - P_1)}{9\ n}\ gr^2 \ldots\ldots (1)$$

where V = the velocity of fall, in centimeters per second,
P_p = the density of falling particle,
P_1 = the density of liquid in which the particle falls,
g = the gravity constant, in centimeter-gram-seconds = 981,
r = radius of particle, in centimeters, and
n = coefficient of viscosity, in poises.

Without appreciable error, values of specific gravity instead of those of density can be substituted for P_p and P_1. Let $P_p = 2.72$, the average specific gravity of the Cobble Mountain Dam material, and $P_1 = 1.00$. Then $P_p - P_1 = 1.72$. Substituting this value in Eq. 1, and also using r, the radius, equal one half d, the diameter,

$$V = \frac{2 \times 1.72}{9\ n}\ 981 \left(\frac{d}{2}\right)^2 = 93.74\ \frac{d^2}{n} \ldots\ldots (2)$$

From Eq. 2,

$$d^2 = \frac{Vn}{93.74}$$

$$d = \sqrt{\frac{1}{93.74}}\ (Vn)^{\frac{1}{2}} = 0.1033\ (Vn)^{\frac{1}{2}} \ldots\ldots (3)$$

Equation 3 was derived with d in centimeters and n in poises. However, it will remain the same for d in millimeters and n in centipoises. The value of n for any temperature can be obtained from tables of coefficients of viscosity; therefore, it only remains to find the value of V by observation during tests for the determination of d; thus,

$$V = \frac{\text{Distance, in centimeters}}{\text{Time, in seconds}} \ldots\ldots (4)$$

The elutriator tests were not only run for preliminary studies but also for the control during the construction. It is true that the Cobble Mountain elutriator carried the gradation to 0.01 mm. and that the distribution of particle sizes finer than 0.01 mm. are important, but the percentage of the latter generally will give the necessary information. If time element is disregarded, there is no reason why additional points could not be obtained for a gradation curve, as suggested by Mr. Morrison.

The author does not agree with the studies and methods suggested by Mr. Morrison because of the following facts in actual construction:

1. The whole of the borrow pit material should be considered in gradation curves for preliminary investigations.

2. Material passing a No. 100 sieve, constituting a 100 per cent curve, has no practical value.

3. There is a definite relation between the core and beach volumes of a dam, and this is not necessarily confined to minus and plus No. 100 sieve quantities of any borrow material.

4. Some of the successful hydraulic fill dam cores contain about 25 per cent particle sizes larger than the No. 100 sieve openings.

5. It should not be taken for granted that material could be wasted. Wasting of fines is sometimes dangerous and impossible not only because of sanitary and industrial restrictions, but because of the involved loss of water during the operation.

6. The rate of settlement of the core will depend not only on various characteristics of the material, but mostly on the speed of construction; and it cannot be predicted by the method suggested which involves "draining off turbid fluid" and ". . . water from the pool must be drawn off . . ." (see 5 above).

7. The term "heavy toes" in hydraulic fill dams conveys the meaning of "rock fill" or coarse fill at the extreme toes of the beaches. These toes have no side bearing of the core, but instead their function is to support the beaches which in turn support, during construction, the liquid or semi-liquid core with heavier specific gravity than water. The effect of the vertical component of the beach material over the sloping or narrowing sides of the cores, designed according to best practice, is nothing to worry about due to more or less automatic control of hydraulic fill construction.

The above reasons are substantiated in detail in the author's writings elsewhere:

"Tunnel Blasting at Cobble Mountain," *The Explosives Engineer*, May, 1930.

"Hydraulic Fill Construction at Cobble Mountain Dam," *Public Works*, June, 1930.

"Constructing Highest Hydraulic Fill Dam," *The Excavating Engineer*, April, 1932.

"Construction of the Cobble Mountain Dam of the Springfield Water Works," *Journal*, New England Water Works Assn., Vol. XLVI, No. 4 (1932).

"Tests for Hydraulic Fill Dams," *Transactions*, Am. Soc. Civil Engrs., Vol. 99, pp. 206–247, 278–294 (1934).

MEASURING THE COMBUSTIBILITY OF FIRE-RETARDANT WOOD

By E. F. Hartman,[1] A. S. Williams[1] and R. C. Bastress[1]

Synopsis

This investigation was made for the purpose of developing satisfactory laboratory tests for measuring the value of fire-retardant treatments for wood, and included a study of all tests known to have been used heretofore. This work involved the performance of nearly 2000 individual experiments. Those tests known as building, splinter, stove, hot rivet and hot plate were found upon preliminary study to offer insufficient promise to warrant detailed investigation. The shaving, crib, timber and fire-tube tests have been studied in detail and summarized data are given covering these.

The shaving test was found to give information on resistance to ignition, tendency to support or communicate flame, and glow. However the test is difficult to duplicate because of variations in dimensions or type of shavings from different species of kiln-dried wood. This test was therefore discarded as unsatisfactory. It appears well suited for a quick plant test on freshly impregnated wood.

The crib test is found to be the best single test since it is easily performed, requires no expensive equipment and gives information on every factor of importance except possibly flame penetration. It is very accurate in its ability not only to detect the presence of but also to give a measure of the amount of untreated material present.

The timber test is the one test which can be used to give information on flame penetration and loss in strength, and it also serves to give some information on the other points. This test, when used in conjunction with the crib test or fire-tube test, gives satisfactory information on all desired points.

The fire-tube test fails to give information on glow, insufficient information on loss in strength or flame penetration and fails to give as complete information as the crib test. It is, however, one of the three most satisfactory test methods studied.

Introduction

The investigation described was undertaken for the purpose of developing satisfactory laboratory methods for measuring the combustibility of fire-retardant wood. Such tests are necessary in specification requirements for commercially treated wood. Various tests have been used heretofore, among which are those known as the building, splinter, Prince's quartz tube, stove, hot rivet, hot plate, fire-tube, shaving, crib and timber tests. The latter three have been used by the New York Department of Buildings for many years. The splinter test[1] was also used as an official test for veneers about 1913, but for some reason was discontinued. In this study particular attention has been given the shaving, crib, timber and fire-tube tests with a view toward modifying them so as to make them more satisfactory. The other tests mentioned were found after preliminary study to offer insufficient promise to warrant a detailed study.

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[1] President; Director, Testing Laboratory; and Chief Engineer, respectively, Protexol Corp., Kenilworth, N. J.

Prince[2] made a study of various test methods and developed an apparatus consisting of an electrically heated quartz tube in which the test specimen was suspended. He used as a measure of inflammability the time necessary for the specimen to ignite at a given temperature. He found the crib and timber tests unsuited for this purpose. He also developed a box type of apparatus in which the specimen was exposed to an electrically heated plate and the gases formed were ignited by a pilot flame.

W. D. Baufield and W. S. Peck[3] made use of an electrically heated apparatus for measuring the ignition temperature of treated and untreated Douglas fir.

Truax and Harrison[4] made a comparison of the crib, timber and shaving tests with the fire tube but made no attempt to modify the three former. They found the apparatus suggested by Prince unsuited for measuring the fire resistance of treated wood.

The British Standards Institution[5] make use of an apparatus very similar to that of Prince for determining combustibility.

Lullin[6] made use of a horizontal tube arrangement for the study of ignition temperatures of both treated and untreated wood and used preheated air as the source of heat. This method, however, is not practical for the type of testing contemplated here.

Hicks[7] used an electrically heated dental furnace and determined the ignition temperature of the evolved gases and the loss in weight of the specimen.

The personal element of the operator has largely been eliminated in this work by the introduction of numerical values of interpretation, based upon time and weight measurements.

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[1] Charles Dahlem, " Fireproofing Wood," unpublished manuscript, New York (1913).

[2] R. E. Prince, "Tests on the Inflammability of Untreated Wood and of Wood Treated with Fire-Retarding Compounds," *Proceedings*, Nat. Fire Protection Assn., Vol. 19, p. 133 (1915).

[3] W. D. Baufield and W. S. Peck, " The Effect of Chemicals on the Ignition Temperature of Wood," *Canadian Chemistry and Metallurgy*, Vol. VI, p. 172 (1922).

[4] T. R. Truax and C. A. Harrison, "A New Test for Measuring the Fire Resistance of Wood," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 973 (1929); also *Proceedings*, Am. Wood Preservers Assn., p. 130 (1930).

[5] British Standard Definitions for Fire Resistance, Incombustibility and Non-inflammability of Building Materials and Structures, published by the British Standards Inst. (1932).

[6] A. Lullin, "Recherches sur les Temperatures D'Inflammation du Bois," Geneva (1925).

[7] C. S. Hicks, " Fireproofing and Antiseptic Treatment of New Zealand Timbers," *The New Zealand Journal of Science and Technology*, Vol. 1, p. 236 (1918).

Information desired in tests must include resistance to ignition, resistance to flame penetration, tendency to support or communicate flame, loss in weight or strength, and tendency to smolder or glow after the removal of the igniting flame. Unfortunately, no single test has been found which gives information on all of these points and it is necessary to make use of at least two tests in order to obtain complete information.

MATERIALS

The material used for test was selected from commercially treated stock containing approximately one-half pound of fire-retardant salts per board foot and had been kiln-dried to a moisture content of 7 per cent ± 1 per cent. Each board was of such dimensions as to provide in most cases sufficient material for shaving, crib, timber and fire-tube specimens, thus forming a better basis for comparison of the different tests.

All tests were carried out in an enclosed hood equipped with mechanical ventilation capable of adjustment so as to give circulation of air at rates varying from approximately 20 to 100 lin. ft. per min. The lowest rate was just sufficient to remove smoke and fumes from the top of the hood. All temperatures were measured by means of a recently calibrated Leeds & Northrup potentiometer temperature indicator.

SHAVING TEST

The shaving test, which is conducted at relatively low temperatures, is an ignition test designed to give information comparable to the flash test in the case of oils. In a study of methods of testing fire-retardant wood Ira H. Woolson[1] investigated the shaving test and found the following objections to it:

1. No limits as to time of burning or burned area can be set. These details must be left to the judgment of the operator.

2. The extreme difficulty in maintaining the same conditions of flame and heat under the shavings.

3. The impossibility of securing a uniform quality of shavings. This last objection he regarded as the most serious and he found it impossible to obtain shavings of uniform size and quality even when the shavings were prepared by the same carpenter using the same plane, set at the same depth of cut. A further objection to the test is that wood as shavings is never used in buildings and he, conse-

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[1] Ira H. Woolson, "A New Method of Testing Fire-Resisting Qualities of Fire-Proofed Wood," *The School of Mines Quarterly*, Columbia University, Vol. 23, p. 248 (1902).

quently, regarded it difficult to interpret the results obtained in the shaving test.

The results obtained in the tests described here lead to the same general conclusions. It was possible to obtain uniformity in the igniting flame by use of a suitable chimney to protect the flame from air currents. However, the wide variation in the dimensions of the shavings from kiln-dried wood could not be overcome and this outstanding handicap makes it apparent that the shaving test is impractical as a means of determining the fire-resistant value of kiln-dried fire-retardant wood.

TABLE I.—RESULTS OF SHAVING TESTS.

Variable flame and draft.

Kind of Wood	Yellow Flame						8-in. Blue Flame						Yellow Flame		
	No Draft		Slow Draft, 20 lin. ft. per min.		Full Draft, 100 lin. ft. per min.		No Draft		Slow Draft		Full Draft		Passed with No Draft	Passed with Slow Draft	Passed with Full Draft
	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed	Passed	Failed			
Chestnut	18	0	18	0	18	0	10	1	11	0	10	0	18	18	18
American walnut	12	0	12	0	11	1	3	0	3	0	3	0	12	12	11
French walnut	1	0	1	0	1	0	..	..	..	..	..	..	1	1	1
English oak	3	0	3	0	3	0	3	0	3	0	3	0	3	3	3
Red oak	7	4	7	0	4	3	5	3	6	3	5	3	7	7	4
Poplar	16	2	16	3	8	8	2	7	2	8	2	12	16	16	8
Birch	3	2	3	1	3	3	0	3	0	3	0	3	3	3	3
Sugar pine	21	1	21	0	18	3	5	3	6	3	3	11	21	21	18
Knotty pine	2	4	2	1	1	5	0	5	0	6	0	7	2	2	1
Yellow pine	1	0	1	0	1	0	..	..	..	..	..	..	1	1	1
Mahogany	1	0	1	0	1	0	1	0	1	0	1	0	1	1	1
Maple	5	5	5	3	2	11	0	10	0	10	0	10	5	5	2

The rules for conducting the test of shavings from fire-retardant wood in accordance with the Code of Ordinances (Building Code) of New York City adopted in 1927, and amended in 1928, are as follows:

Shaving Test.—A mass of shavings cut fairly thick from the outside and interior of sticks of the treated wood are to be tested separately. These shavings shall be placed to a depth of 2 in. in a metal vessel 12 in. square, the bottom of which shall consist of a wire screen of ½-in. mesh. The shavings shall be packed down moderately to reduce the air spaces. A bunsen yellow flame shall then be placed beneath the receptacle so that the flame is in contact with the shavings. After 25 sec. the flame shall be removed. The flame at no time should show higher than 6 in. above the top of the bed of shavings and the shavings should not be consumed in less than 5 min.

Test Data:

In this investigation a total of 529 separate tests was made upon 183 samples of shavings from 12 different species of kiln-dried wood.

The conditions under which the tests were made were as follows: (*a*) no draft; (*b*) slow draft (20 lin. ft. per min.); (*c*) full draft (100 lin. ft. per min.), all with a yellow flame of a bunsen burner as specified above. Since the height of flame, its distance from the shavings, and the length of flame impinging upon the shavings are not specified, the following interpretation was given to these factors: Distance from top of bunsen burner to wire screen $3\frac{1}{4}$ in., total height of flame $4\frac{3}{4}$ in., length of flame impinging upon shavings $1\frac{1}{2}$ in. The latter dimension was sufficient to maintain contact between flame and shavings even when the flame was subjected to momentary horizontal air currents. The temperature of this flame at the zone of contact with the shavings at the end of the 25-sec. exposure varied from 1075 to 1125 F. and averaged about 1100 F. as measured by a 14-gage chromel-alumel thermocouple.

TABLE II.—EFFECT OF DRAFT ON RATE OF BURNING.
Standard yellow flame.

Kind of Wood	No Draft		Slow Draft		Full Draft	
	Percentage Burned	Time Burned, min.:sec.	Percentage Burned	Time Burned, min.:sec.	Percentage Burned	Time Burned, min.:sec.
American walnut	0	0	0	0	20	....
	0	0	0	0	100	2:00
	0	0	0	0	5	....
	Smoke[a]	2:00	Smoke	1:40	Smoke	2:30
Red oak	0	0	0	0	10	....
	Smoke	4:00	Smoke	5:00	100	22:00
	Smoke	1:30	Smoke	1:20	100	4:50
Maple	30	5:10	..	....	70	3:00
	100	1:45	100	1:40	100	1:20
	Smoke	1:30	Smoke	1:20	100	4:50
	0	0	0	0	100	5:55
	100	7:15	100	9:50	100	5:03
	100	2:05	..	....	100	1:40
	100	10:35	..	....	100	9:10
Poplar	0	0	0	0	100	2:00
	10	....	10	....	10	....
	0	0	0	0	100	2:30
	0	0	0	0	100	2:55
Birch	0	0	0	0	33	2:00
	100	2:10	100	1:50	100	1:40
	100	1:50	100	2:05	100	1:30
	100	5:20	100	3:50	100	3:10
	100	4:00	100	3:55	100	2:50
	100	2:28	100	2:45	100	2:25
Knotty pine	0	0	0	0	100	2:00
Sugar pine	0	0	0	0	5	....

[a] No flame produced but sufficient smoke to indicate some combustion.

In some cases where there was no ignition of the shavings with the standard yellow flame, the use of a 4-in. (approximate temperature 1450 F.) or 8-in. (approximate temperature 1550 F.) blue flame was introduced in order to determine whether the draft had any effect upon the burning rate or blaze height once ignition of the shavings had taken place, for a difference, if any, could only be noticed when at least a part of the shavings was consumed. Or, in other words, samples on the border line of passing the usual test were the ones where the effect of draft was most noticeable. The use of the blue flame was simply an attempt to increase the number of burning samples. The results of these tests are summarized in Table I.

A study of this table (see last three columns) shows that of those samples which passed using no draft all passed using slow draft (20 lin. ft. per min.), and with full draft (100 lin. ft. per min.) the following percentages passed: American walnut 91.67; red oak 57.14; poplar 50; sugar pine 85.7; knotty pine 50 and maple 40. Or for all samples which passed with no draft 17.4 per cent failed with full draft. In a number of cases, observation was made of the effect of draft upon the rate of burning and the percentage of the sample burned. These results are given in Table II.

It is clear from these results that the effect of full draft is quite pronounced. In eight instances the specimen passed using no draft and slow draft but failed completely when full draft was used. In other instances the difference in rate of burning is very evident, full draft hastening the burning of the sample. A study of Table II also shows that in some cases only a partial burning of the sample occurs. It is sometimes noticed that a small hole will burn through the layer of shavings and then the blaze disappears. Because of such occurrences the percentage of shavings burned was noted, and in these tests there were instances of from 5 per cent to 95 per cent as well as the zero and 100 per cent portions burned. Such occurrences were noticeable particularly when it was necessary to resort to a blue flame to obtain ignition. It is seen that the condition of partial burning occurs most frequently when full draft is used.

During the progress of this investigation it was apparent that in the cases where a blaze was produced by the burning shavings the height of the blaze was considerably greater when full draft was used. As a result of over 200 observations on this particular point it was found that with full draft the height of the blaze from the burning shavings was from 20 to 30 per cent greater than when no draft or slow draft was used.

Another factor affected by draft conditions is the temperature of the burning shavings. This was measured in a number of cases by placing a thermocouple immediately above the upper surface of the shavings in the center of the metal basket during the test and measuring the temperature as the burning progressed. In this connection it should be pointed out that there is sometimes a variation in the manner in which the shavings take fire and burn. Although the igniting flame was always placed under the center of the shavings, it was found that sometimes the shavings burned around the edge first, or at one side, instead of in the center. In such cases it was necessary to shift the position of the thermocouple relative to the basket so as to get as nearly as possible a correct temperature reading. The results of 70 tests made to determine the effect of draft on the temperature of the burning shavings are as follows:

	Average Temperature, deg. Fahr.		
Kind of Wood	No Draft	Slow Draft	Full Draft
Birch	726	768	872
Mahogany	565	645	705
Poplar	442	603	690
Maple	455	362	763
Sugar pine	560	550	748
Average	550	586	756

In all cases it was found that the temperature of the burning shavings was greater when full draft was used.

It was felt that the temperature of the hood might have some effect upon the shaving test, consequently 137 observations on this factor were made. The temperature of the hood in these tests varied from 15 C. (59 F.) to 50 C. (122 F.) and no effect was noticeable. This is undoubtedly due to the fact that the shavings were not in the hood long enough before test to allow them to reach the same temperature as the hood and, since in practice such is the case, no further tests were made.

One additional factor which seemed to have a bearing upon the shaving test is the nature, size, etc., of the shavings. In preparing shavings for test it is found that in some cases the shavings are short, slightly curled, with dimensions about $\frac{1}{32}$ by $\frac{1}{2}$ by 1 in. Such shavings are usually obtained from (*a*) the edge or surface of the wood, (*b*) all oak, walnut, chestnut and mahogany. On the other hand, long curly, spiral-like shavings are obtained, dimensions about $\frac{1}{32}$ by 1 in. by several inches to 3 ft. long from such woods as poplar, birch, pine and maple. It was found that the short shavings packed more closely in the basket were more resistant to ignition, burned more slowly, produced a shorter blaze and a lower temperature than the longer curly shavings, while the latter, because of their springiness, require more packing to obtain the same quantity of shavings. A 2-in. depth of short shavings corresponds to a 3 or 4-in. depth of curly shavings.

The pressure of the gas supplying the burner was maintained at from 3.8 to 4 in. of water at all times.

Although not mentioned in the Building Code specifications some form of chimney or windshield is necessary to prevent the igniting flame being swept about by air currents. It was found that during a 25-sec. exposure, even with no draft, the flame was often actually in contact with the shavings less than 15 sec., consequently it was necessary to repeat the test. Furthermore, with full draft the contact between the flame and shavings during a 25-sec. exposure averaged less than 10 sec. Accordingly, a large number of tests was made using a chimney 3 in. in diameter and 6 in. in height (Eimer & Amend No. 19392) with the result that there was practically no flickering of the flame with no draft or slow draft, and in the case of full draft the flame was in contact on an average of between 15 and 20 sec. during a 25-sec. exposure. In some cases using full draft it was necessary to place the top of the bunsen burner as close as 1½ in. to the wire screen in order to provide constant contact between the flame and shavings.

Conclusions (*Shaving Test*):

This study brought out the following regarding the shaving test:

1. The results with slow draft were practically no different from those with no draft.

2. The effect of full draft was pronounced in causing (*a*) the failure of approximately 17 per cent of the samples which passed using no draft and slow draft; (*b*) an increased rate of burning of the shavings; (*c*) an increase of 20 to 30 per cent in the height of the blaze of burning shavings; (*d*) flickering of flame resulting in irregular contact between igniting flame and shavings; (*e*) increase of approximately 50 per cent in the temperature of the burning shavings; (*f*) spotty or partial burning of shavings.

3. The shaving test, although a valuable plant test, is unreliable and of no value on kiln-dried material.

Crib Test

The idea of employing a number of small pieces of material (of approximately ½ by ½ by 6 in.) arranged in equally spaced tiers was suggested early in the history of fire-retardant wood. In 1887, Boudin and Donny[1] of the University of Ghent devised a test consisting of four sticks, approximately ½ in. square and 16 in. long, placed in a revolving cylinder heated by a row of 5 bunsen burners. Later, they used 36 sticks, approximately ½ in. square and 8 in. long, arranged in 6 tiers of 6 sticks each, placed upon a heavy wire gauze. As a source of heat they used small bundles of splints weighing 35 g. each.

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[1] Boudin and Donny, "Rapport sur les procedes destines a assurer l'inflammabilite des bois," Ghent, Belgium (1887).

In 1902, Charles L. Norton[1] used a similar test which he described as the "cob house" test. The sticks ($\frac{3}{4}$ by $\frac{3}{4}$ by 6 in.) were arranged in tiers, those of alternate layers being placed at right angles, and this test seems to have served as a basis for the crib test now used by the New York Department of Buildings.

In the tests referred to above it was customary to use an equal number of sticks in each layer or tier. The records show, however, that during the period around 1912 the New York Department of Buildings specifications called for 10 sticks arranged 4 tiers high with 4 sticks in the bottom layer, 3 in the second, 2 in the third and 1 stick on top. Later the number of sticks was increased to 20 and these were placed in 5 tiers of 4 sticks each as described in the present specifications of the Building Department as follows:

> *Crib Test.*—Twenty samples are to be prepared, each $\frac{1}{2}$ in. square and 6 in. long. These shall be built up on a ring support to form a crib work 5 tiers high, 4 sticks to a tier, making the crib 6 by 6 in. and approximately $2\frac{1}{2}$ in. high. The crib shall be set 6 in. above a bunsen burner to which the crib shall be exposed for a period of 1 min. at a temperature approximating 1200 F. The flame must not persist more than 20 sec. after the bunsen burner is removed, nor the glow last more than 30 sec. The tendency of the flame to spread from stick to stick must also be noted.

The statement that "the tendency of the flame to spread from stick to stick must also be noted" is not sufficiently definite to permit classification of a given sample as having failed or passed the test.

In carrying out the investigations described here it was proposed to establish experimentally a basis for the conditions of the test. It appeared desirable to obtain information upon the following:

1. The effect of air velocity or draft in the hood.
2. The effect of changing gas pressure.
3. The method of establishing the temperature of the igniting flame.
4. The effect of moisture content of wood.
5. The minimum and maximum number of sticks or tiers which should be used.
6. The relation between the time of exposure to the igniting flame and the percentage loss in weight.
7. The relation of height of blaze from the burning crib and its temperature to the fire-resistive qualities of the wood.

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[1] *Report No. 1*, "Fire-Proof Wood . . . . So-called," Insurance Engineering Experiment Station, Boston, Mass., August, 1902.

8. The relation of duration of flame or blaze after removal of the igniting flame to the fire-resistive qualities of the wood.

9. The relation of duration of glow to the fire-resistive qualities of the wood.

10. The relation of the percentage loss in weight to the fire-resistive qualities of the wood.

1. *Effect of Air Velocity or Draft.*—Tests were conducted at air velocities of 20 and 100 lin. ft. per min. through the hood. The flame was protected from air currents by the use of a 3 by 6-in. chimney. With an air velocity of 100 lin. ft. contact between the flame and the specimen was maintained for less than one-fourth of the time and even then the flickering of the flame was so great that the bottom tier of sticks was barely charred on the bottom face. This condition was not materially changed by use of the chimney. It was found, however, that if exposure of the specimen to the flame, by holding the burner close to the bottom tier, was continued until ignition occurred then the higher air velocity caused a greater extent of burning. For uniform results, therefore, the use of such a high air velocity made tests worthless as a basis of measurement or comparison.

TABLE III.—AVERAGE RESULTS SHOWING THE EFFECT OF A FLAME OF APPROXIMATELY 1200 F. FOR 1 MIN. UPON OVEN-DRIED CHESTNUT MATERIAL.

Twenty pieces per crib.

Number of Tests	Tier	Loss in Weight, per cent	Percentage of Whole Lost by Each Tier	Blaze Height, in.	Continued Flame, sec.	Glow, sec.	Number of Pieces Burning
2	No. 1	6.38	37.70	0	..	..	..
	No. 2	5.23	31.40				
	No. 3	3.19	19.90				
	No. 4	1.02	6.26				
	No. 5	0.81	5.62				
Total	....	3.33	....	0	0	4	0

2. *Effect of Changing Gas Pressure.*—It is of course obvious that the size of flame as well as its temperature are both affected by the pressure of the gas supplying the flame. Consequently, the tests described here were conducted at a gas pressure which varied only from 3.5 to 3.75 in. of water. Under such conditions uniform results were obtained.

3. *Temperature of the Igniting Flame.*—According to the specifications of the New York Department of Buildings, the crib is to be placed above a bunsen burner and exposed to a temperature approximating 1200 F. However, no method for determining or establishing that temperature is prescribed. In these tests the flame

temperature was measured by use of an 8-gage iron-constantan thermocouple. A flame adjusted so that such a thermocouple registers 1200 F. ± 25 F. is approximately 7.5 in. high with a slight indication of yellow just above the green inner cone of the burner.

4. *Effect of Moisture upon Loss in Weight.*—In order to determine the effect of moisture upon the crib test, sufficient material to prepare two 5-tier cribs was dried to constant weight in an oven at 105 C. (220 F.). This material, when tested, gave the results shown in Table III.

Since the average percentage loss for ordinary kiln-dried material containing approximately seven per cent moisture, as shown by the averages for 20 pieces in Table V, is 2.69 per cent, the presence of this amount of moisture has a slight influence in reducing the percentage loss in weight. The oven-dried material takes up moisture very rapidly and this requires rapid work in making tests on such material.

5. *Minimum and Maximum Number of Sticks or Tiers that Should be Used.*—Since one of the objects of the crib test is to measure the tendency of the material to communicate flame and build up temperatures, it is obvious that a single tier of four sticks would not give this information. Furthermore, since the igniting flame is capable of reaching from 1 to 1½ in. above the iron ring support, a sufficient number of tiers should be used which would utilize the entire flame available. These considerations lead to the conclusion that two tiers or eight sticks would be the minimum number from which the desired information on communication of flame, etc., could be obtained.

It might appear that the length of flame impinging upon the specimen, that is the upper 1½ in., would determine the maximum number of tiers which could be used. However, it is found that a flue or chimney effect is introduced which makes it possible for the flame to reach a height greater than three tiers. This is shown by the appearance of the blaze above the top of the crib, and also by the loss in weight of the top tier even when seven tiers were used.

In order to determine the effect of the flame upon cribs of varying numbers of sticks, a number of tests were conducted using 8, 12, 16, 20, 24 and 28 sticks per crib. The sticks were placed in tiers of 4 pieces each and alternate layers were placed at right angles. Each tier in some cases was weighed separately before and after exposure to the flame and the loss in weight calculated. These data together with information on other factors such as the height of the blaze formed by the burning crib, the duration of flame and glow after removal of the igniting flame, etc., are given in Tables IV and V.

Comparable data can be obtained only when uniformly treated material is used. Variations in kind or extent of treatments or in species of wood introduce other variables which render conclusions more difficult. Chestnut was selected because it offers visible evidence on the thoroughness of the treatment and thus the likelihood of error in interpreting results was obviated.

TABLE IV.—AVERAGE RESULTS SHOWING THE EFFECT OF EXPOSURE OF THE CRIB OF CHESTNUT MATERIAL TO A FLAME AT APPROXIMATELY 1200 F. FOR 1 MIN.

Number of Tests	Number of Pieces per Crib	Loss in Weight, per cent	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
8	8	3.91	2	0	2	0
8	12	4.38	2	2+	2−	..
7	16	3.00	1	1+	2+	0

TABLE V.—AVERAGE RESULTS SHOWING THE EFFECT ON CHESTNUT MATERIAL OF A FLAME OF APPROXIMATELY 1200 F. FOR 1 MIN. UPON THE DIFFERENT TIERS OF THE CRIB.

Number of Tests	Number of Pieces per Crib	Tier	Loss in Weight, per cent	Percentage of Whole Lost by Each Tier	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
5	20	No. 1	5.32	40.44	1	..	..	..
		No. 2	4.08	29.86				
		No. 3	2.22	16.69				
		No. 4	1.25	9.57				
		No. 5	0.49	3.51				
Average			2.69	.....	1	0	3	0
2	24	No. 1	6.71	36.08	1	..	..	..
		No. 2	5.07	26.34				
		No. 3	3.18	15.95				
		No. 4	2.15	10.69				
		No. 5	1.34	6.82				
		No. 6	0.84	4.04				
Average			3.30	.....	1	0	4	0
2	28	No. 1	6.32	35.65	..	..	..	..
		No. 2	5.30	29.87				
		No. 3	3.44	18.95				
		No. 4	1.95	11.06				
		No. 5	1.10	6.56				
		No. 6	0.52	3.22				
		No. 7	0.28	1.62				
Average			2.64	.....	0	0	3	0

SUMMARY

Number of Tiers per Crib	Average Loss per Tier, per cent	Average Loss of Crib, per cent
2	1.96	3.91
3	1.46	4.38
4	0.75	3.00
5	0.53	2.67
6	0.36	3.30
7	0.38	2.64

From these data it is found that the percentage loss in weight per tier becomes practically constant with 6 tiers. This number would thus represent the maximum number of tiers necessary for this test.

6. *Relation Between the Time of Exposure and the Percentage Loss in Weight.*—A number of tests were conducted at 1200 and 1700 F. using 12 pieces of treated chestnut per crib and varying the time of exposure from 1 to 4 min., as reported in Table VI. Similar tests for 1 and 2 min. at 1200 F. using 5-ply veneer are reported in Table VIII. In order to prevent burned or broken sticks from falling off the iron ring support, the crib was built up on $\frac{1}{2}$-in. mesh wire gauze.

The results obtained in using both 1200 F. and 1700 F. flame indicate a constant relationship between time of exposure and percentage loss in weight.

TABLE VI.—AVERAGE RESULTS SHOWING THE RELATION OF TIME OF EXPOSURE TO LOSS IN WEIGHT FOR CHESTNUT MATERIAL.

Twelve pieces per crib.

Number of Tests	Time of Exposure, min.	Loss in Weight, per cent	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
FLAME OF 1200 F.						
3	1.0	2.07	0	0	0	0
3	1.5	3.42	1+	0	0	0
3	2.0	4.88	2−	0	0	0
3	2.5	5.86	2	0	2	0
3	3.0	7.13	2	0	3	0
3	3.5	8.72	2	0	3	0
3	4.0	10.26	2	0	4	0
FLAME OF 1700 F.						
3	1.0	8.94	6.7	0	14	0
3	1.5	14.71	7.3	15	13	2
3	2.0	20.40	7.7	16	21	2
3	2.5	25.18	8.0	32	8	2
3	3.0	29.79	8.3	45	57	3

As a final check upon the correct time of exposure to employ in the crib test, two series of tests were performed in which a definite amount of untreated wood was introduced. In one series one stick of untreated chestnut was included with eleven sticks of treated material, the untreated stick being placed in the bottom layer next to the center. In the other series cribs were prepared from 5-ply veneer panels $\frac{1}{2}$ in. thick containing treated and untreated material. The results are given in Tables VII and VIII.

7. *Relation of Height of Blaze from the Burning Crib and Its Temperature to the Fire-Resistive Qualities of the Wood.*—In this series of tests the height of the blaze formed by the burning crib was estimated by placing a metal rule just behind the crib. The tempera-

ture of this blaze was also measured by means of an 8-gage iron constantan thermocouple placed $\frac{1}{2}$ in. above the center of the top layer of the crib.

It was found that for a 1-min. exposure to a flame of 1200 F. the blaze temperature averages about 800 F. and for a 2-min. exposure a temperature of about 1000 F. is reached, while the height of the blaze averages slightly less than 2 in. for a 1-min. exposure and slightly more than 2 in. for a 2-min. exposure. It is probable that the time of exposure has more effect upon the temperature reached than upon the height of the blaze. These results are shown in Table IX.

TABLE VII.—THE EFFECT OF THE PRESENCE OF UNTREATED CHESTNUT MATERIAL UPON THE TEMPERATURE PRODUCED AND LOSS IN WEIGHT.

Twelve pieces per crib.

Sample	Time of Exposure, min.	Loss in Weight, per cent	Percentage of Untreated Material Present	Temperature at Top of Crib, deg. Fahr.	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
No. 57	1	7.57	7.32	900	5	135	0	1
No. 58	1	8.94	7.65	820	5	95	0	1
No. 59	1	8.25	7.91	950	4	94	0	1
No. 60	1	9.02	7.86	890	5	98	0	1
No. 61	1	8.38	8.08	875	5	78	0	1
Average	1	8.43	7.76	887	5	100	0	1
No. 62	1	95.02	100.0	1360	30	275	1070	12
No. 63	1	94.02	100.0	1260	30	295	1120	12
Average for all untreated	1	94.52	100.0	1310	30	285	1095	12

TABLE VIII.—AVERAGE RESULTS SHOWING THE EFFECT OF THE PRESENCE OF UNTREATED MATERIAL UPON THE FIRE RESISTIVE QUALITIES OF VENEER.

Twelve pieces per crib.

Number of Tests	Time of Exposure, min.	Loss in Weight, per cent	Percentage of Untreated Material	Dimensions, in., and Treatment[a]				Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
				Core	Cross-Banding	Face	Back				
2	1	5.04	23.10	$\frac{1}{4}$T	$\frac{1}{16}$T	$\frac{1}{20}$U	$\frac{1}{16}$U	3	6	0	1
3	2	9.81	23.10	$\frac{1}{4}$T	$\frac{1}{16}$T	$\frac{1}{20}$U	$\frac{1}{16}$U	4+	15	13	1
3	2	32.55	38.92	$\frac{11}{32}$T	$\frac{1}{16}$U	$\frac{1}{32}$U	$\frac{1}{16}$U	15	102	147	12
2	1	8.03	27.80	$\frac{11}{32}$T	$\frac{1}{16}$U	$\frac{1}{32}$U	$\frac{1}{16}$T	7	51	107	2
3	2	16.69	27.80	$\frac{11}{32}$T	$\frac{1}{16}$U	$\frac{1}{32}$U	$\frac{1}{16}$T	9	118	62	4
4	1	7.13	65.86	$\frac{1}{4}$U	$\frac{1}{12}$T	$\frac{1}{28}$U	$\frac{1}{28}$U	4	42	16	0
6	2	77.90	65.86	$\frac{1}{4}$U	$\frac{1}{12}$T	$\frac{1}{28}$U	$\frac{1}{28}$U	20	387	821	12

[a] T = treated; U = untreated.

When definite measured amounts of untreated material are introduced, either as untreated sticks or as part of veneer, the effect is noticeable both upon the temperature and height of the blaze, as shown in Tables VII and VIII. The greater effect is noticed upon the blaze height which changes from less than 2 in. to nearly 5 in. for a 1-min. exposure, while the blaze temperature increases approximately 100 deg. with the introduction of slightly more than 8 per cent of untreated material.

8. *Relation of Duration of Flame or Blaze After Removal of the Igniting Flame to the Fire-Resistive Qualities of the Wood.*

9. *Relation of Duration of Glow to the Fire-Resistive Qualities of the Wood.*—Examination of the data given above indicates that the duration of flame and glow both give a measure of the fire-resistive qualities of the material. This is particularly noticeable where known quantities of untreated material are present as shown in Tables VII and VIII. While it might be expected that flame and glow are capable of giving qualitative information on the presence of untreated material, it is found that they are not capable of giving a quantitative measure of untreated material.

TABLE IX.—THE RELATION BETWEEN LOSS IN WEIGHT AND THE TEMPERATURE PRODUCED AT THE TOP OF THE BURNING CRIB FOR CHESTNUT MATERIAL (TEMPERATURE OF IGNITING FLAME 1200 F.).

Twelve pieces per crib.

Sample	Time of Exposure, min.	Loss in Weight, per cent	Temperature at Top, deg. Fahr.	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
No. 37	1	2.85	760	2	0	3	0
No. 38	1	2.55	800	2+	0	3	0
No. 39	1	2.92	850	2	0	3	0
No. 40	1	2.38	740	2	0	3	0
No. 41	1	2.48	750	2	0	3	0
No. 42	1	2.93	800	2	0	3	0
No. 43	1	2.40	790	2	0	3	0
No. 44	1	2.38	830	1+	0	3	0
No. 45	1	3.16	825	2	0	3	0
No. 46	1	2.43	790	1+	0	3	0
Average 1 min	....	2.65	794	2	0	3	0
No. 47	2	7.18	825	1	0	5	0
No. 48	2	5.89	920	2	0	4	0
No. 49	2	8.16	960	3	0	4	0
No. 50	2	8.86	1020	2	0	4	0
No. 51	2	7.50	1035	2	0	5	0
No. 52	2	9.17	1080	3	0	5	0
No. 53	2	7.93	1080	2	0	4	0
No. 54	2	7.83	1025	2	0	4	0
No. 55	2	8.37	1040	3	0	4	0
No. 56	2	8.23	1010	3	0	4	0
Average 2 min	....	7.91	999.5	2+	0	4+	0

10. *Relation of the Percentage Loss in Weight to the Fire-Resistive Qualities of the Wood.*—The results obtained throughout this investigation show a fairly uniform relationship between the percentage loss in weight and the fire-resistive qualities of the material. This is particularly brought out by the results obtained by introducing

definite amounts of untreated material as shown in Tables VII and VIII.

From the results of these tests it is evident that an exposure of 1 min. at 1200 F. does not give as correctly the percentage of untreated material present as is given by a 2-min. exposure. This is shown clearly by the percentage loss in weight of veneer containing both treated and untreated material. Particularly is this noticeable in the data given in Table VIII. Here it is apparent that a 1-min. exposure gives little if any evidence of the presence of over 65 per cent untreated material, whereas a 2-min. exposure gives a quite accurate measure of the amount of untreated material present. The total percentage loss in weight is slightly greater than the amount of untreated material present due to the slight charring of the treated material which is to be expected under these conditions. Furthermore, in carrying out these tests it was observed that the blaze from the burning crib reached its maximum height at the end of $2\frac{1}{4}$ to $2\frac{1}{2}$ min., and finally in those cases where the temperature produced by the burning crib was recorded it was found that it also reached a maximum at from $1\frac{3}{4}$ to 2 min. All of these factors, therefore, indicate that the exposure to the igniting flame in the crib test should be for a minimum period of 2 min. instead of 1 min. as at present specified by the New York Department of Buildings.

TABLE X.—RESULTS OF EXPOSURE OF CRIB OF TWELVE PIECES OF WHITE PINE TO A FLAME OF 1200 F. FOR 2 AND 3 MIN.

Sample	Time of Exposure, min.	Loss in Weight, per cent	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
No. 1	2	4.02	2	0	0	0
No. 2	2	4.08	2	0	0	0
No. 3	2	3.74	2	0	0	0
No. 4	2	4.48	4	0	0	0
No. 5	2	4.42	2	0	0	0
Average 2 min.	....	4.15	2.4	0	0	0
No. 1	3	6.87	2	0	0	0
No. 2	3	6.68	2	0	0	0
No. 3	3	5.86	2	0	0	0
No. 4	3	6.58	2	0	0	0
No. 5	3	6.12	2	0	0	0
Average 3 min.	....	6.42	2	0	0	0

In order to give the results of this investigation a more general application, information was obtained on a soft wood by conducting a series of tests using treated white pine at exposures of 2 and 3 min. The results obtained are given in Table X.

These results when compared with results obtained for chestnut at an equal time of exposure as shown in Table VI indicate a slightly greater resistance in favor of the white pine. However, the performance of the two woods shows a rather close agreement.

Conclusions (Crib Test):

The results obtained in the foregoing critical study indicate that:

1. The velocity of the air in the hood during the test has a marked effect upon the results. Velocities greater than 20 lin. ft. of air per minute cause flickering or waving of the flame from side to side to such an extent as to preclude uniformity of results, even when the flame is protected by means of a chimney 3 in. in diameter and 6 in. high.

2. A variation in gas pressure supplying the igniting flame of more than $\frac{5}{10}$ in. of water gave pronounced difference in the flame temperature and in the percentage loss in weight in the crib test.

3. The temperature of the igniting flame should be controlled by means of a thermocouple placed in the center of the plane of the bottom of the crib.

Tests conducted at a temperature of 1700 F. show a greater percentage loss in weight per minute of exposure than tests at 1200 F. An exposure at 1700 F. for 1 min. gives a loss in weight of approximately 9 per cent, corresponding to an exposure of about 3.5 to 4 min. at 1200 F. Duration of flame and glow as well as height of blaze and number of pieces burning are all greater when a 1700 F. flame is used. It appears that equally satisfactory results are obtainable at either temperature.

4. The moisture content of the wood has little appreciable effect up to 7 per cent moisture, the maximum present in these tests. The loss in weight is slightly greater for oven-dry wood, but the difference is less than 1 per cent.

5. The number of tiers per crib should be greater than two so as to utilize all of the flame available, when adjusted to give a temperature of 1200 F. at the bottom of the crib. Furthermore, information on the tendency of the fire to spread from tier to tier can be obtained only if two or more tiers are used. On the other hand the maximum number of tiers is limited to those actually affected by the igniting flame. In the experiments described, the number of tiers varied from 3 to 7 and in no case was a loss in weight per tier greater than 1 per cent for those tiers above the fifth. It would seem impractical, therefore, to use more than 5 tiers per crib. In all cases the percentage loss in weight is greater in the bottom or No. 1 tier and amounts to about 5 or 6 per cent. Since approximately 85 per cent of the total loss in weight is confined to the 3 lower tiers, it appears

that satisfactory results can be obtained by using 3 tiers and less time is necessary in preparing the samples.

6. The time of exposure to the igniting flame should be at least 2 min. This is shown particularly in the results from cribs made up of veneer containing known amounts of untreated material either as core, crossbanding or surface layers. An exposure of 1 min. is sufficient when the untreated material is on or near the surface of the test pieces, but when near the middle 2-min. exposure is necessary to indicate its presence.

The factors which give information of value in the crib test are:

1. Percentage loss in weight which gives an indication of the loss in strength or structural qualities. It also gives a measure of the amount of combustible material present as well as indicating the ignition resistance of the material.

2. The height of blaze from the burning crib gives a measure of the degree of combustibility of the material present, also the tendency of the material to ignite, to communicate fire and build up temperature.

3. Duration of flame indicates the presence of combustible material qualitatively but not quantitatively, also the tendency to spread or communicate fire.

4. Duration of glow indicates the tendency of the material to spread fire by smoldering and may also indicate the tendency to form carbon monoxide.

5. The temperature at the top of the burning crib indicates the amount of combustible material present, also the tendency to spread fire.

Observation of these factors showed that all are important. However, it is thought that the percentage loss in weight, the continued flame and glow and height of blaze are of most importance in supplying information concerning the fire-resistive qualities of the wood being tested.

TIMBER TEST

The timber test was devised by Ira H. Woolson of Columbia University Testing Laboratory. The dimensions of the test specimens as at first established were 1 in. square and 12 in. long, but were subsequently changed to $\frac{3}{4}$ by $1\frac{1}{2}$ by 12 in. His method involved testing two pieces simultaneously by placing them over a 6-in. gas crucible furnace for 2 min. at a temperature of 1700 F. following which the length of time during which they continued to flame or glow was noted. Afterwards they were cut through transversely at the point of smallest cross-section, tracings were made of the unburned portion and the area determined by the use of a planimeter. The particular temperature of 1700 F. was chosen because it was the temperature given by the New York Building Code as approximately that of a burning building and generally recognized as the conflagration temperature.

The present specifications for the timber test as prescribed by the New York Department of Buildings are as follows:

> *Timber Test.*—Two samples ¾ by 1½ in. in cross-section and 12 in. long shall be laid side by side across the top of a gas crucible furnace with a pyrometer between them. The specimens shall be subjected to a flame of 1700 F. for 2 min., the test pieces shall then be removed, and the time of duration of flame and glow observed. The sticks shall then be cut through the middle at the most burned section and the area of the unburned wood measured with a planimeter. The flame must not persist longer than 15 sec. nor the glow longer than 20 sec., and in the case of hardwoods, the unburned area should not be less than 55 per cent, nor in the case of softwoods less than 45 per cent.

These specifications do not entirely remove the personal element of the operator, for example, in the scraping away of the charred portion. Since the line of demarcation between charred and uncharred wood is distinguished by a color change which is gradual and not abrupt, the amount of material to be scraped away is based entirely upon the judgment of the operator. Furthermore, the point of smallest cross-section is likewise subject to the judgment of the operator and in many instances it is practically impossible to determine this visually.

The object of this part of the study was to ascertain whether the timber test could be made to give additional information which would be free from the above disadvantage and yet give true and comparable data on the fire-retardant value of the specimen. The loss in weight suggested itself as in line with usual methods for determining combustion loss. The use of the percentage loss in weight removes an early criticism of this test made by C. F. McKenna.[1] Accordingly experiments were made upon treated and untreated specimens from eight species of hard and four species of soft woods involving over 500 separate tests. The tests involved exposure of the weighed specimen to the flame of a 6-in. gas crucible furnace[2] at a temperature of 1700 F. ± 25 F. for periods of 1, 2 and 3 min. The specimens were tested in pairs having their widest faces (1½ in.) down and their inner sides (¾ in.) in close contact immediately over the thermocouple measuring the temperature of the furnace. The outer sides of the specimens were flanked closely by fire brick as is done in official tests for the N. Y. Building Department. An additional fire brick was placed at the ends of the other two bricks at a distance of 2 in. from the inside of the furnace wall so that ends of the specimens could rest against it. In this way all specimens were in the same relative position over the furnace, thus exposing an equal area of each specimen to the fire.

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[1] C. F. McKenna, "A Proposed Method of Testing Wood Treated to Resist Fire," *Journal*, Am. Chemical Soc., April, 1903, p. 406.

[2] No. 2 Melter manufactured by the American Gas Furnace Co., Elizabeth, N. J.

TABLE XI.—AVERAGE RESULTS OF TIMBER TESTS EXPOSED TO A FLAME OF 1700 F.

Kind of Wood	Number of Specimens Tested	Treatment[a]	Time of Exposure, min.	Loss in Weight, per cent				Percentage of Area Unburned				Percentage of Area Burned	Flame, sec.	Glow, sec.
				Avg.	Max.	Min.	Diff.	Avg.	Max.	Min.	Diff.			
Ash	8	U	2	34.8	39.4	29.9	9.5	44.6	50.7	39.1	11.6	55.4	74	242
	10	T	2	17.6	20.0	15.6	4.4	64.4	68.7	61.7	6.8	35.6	9	0
Birch	6	U	2	40.7	46.8	37.0	9.8	36.6	50.8	8.9	41.9	63.4	372	236
	8	T	2	18.5	15.7	19.9	4.2	66.6	72.5	58.9	13.6	33.4	5	0
Chestnut	8	U	1	23.1	27.7	19.2	8.5	62.4	72.7	42.0	30.7	37.6	68	0
	8	U	2	42.2	49.7	36.8	12.9	32.2	39.1	21.4	17.7	67.8	73	5
	10	U	3	60.8	65.5	54.0	11.5	10.8	28.5	0.0	28.5	89.2	210	45
	8	T	1	15.4	16.6	13.8	2.8	67.9	72.8	64.8	8.0	32.1	4	0
	10	T	2	22.4	23.4	19.8	3.6	55.1	60.5	51.3	9.2	44.9	4	0
	10	T	3	32.4	37.5	26.9	10.6	34.5	42.7	21.3	21.4	65.5	6	0
Douglas fir	7	U	2	30.9	35.0	28.0	7.0	57.1	63.1	51.2	11.9	42.9	44	20
	8	T	2	18.9	21.4	17.3	4.1	66.1	68.5	64.0	4.5	33.9	9	0
Maple	8	U	2	53.2	75.1	44.4	30.7	21.4	38.2	0.0	38.2	78.6	409	864
	10	T	2	21.2	22.4	20.0	2.4	58.3	64.0	47.8	16.2	41.7	13	0
Red oak	6	U	2	39.4	41.3	36.7	4.6	46.5	55.0	40.3	14.7	53.5	251	83
	10	T	2	19.9	21.0	18.5	2.5	59.9	63.8	53.4	10.4	40.1	11	0
White oak	6	U	2	49.5	56.1	45.3	10.8	21.3	38.6	7.6	31.0	78.7	301	1784
	10	T	2	19.0	20.8	16.7	4.1	70.2	77.5	67.0	10.5	29.8	10	0
Walnut	8	U	2	35.4	40.7	32.7	8.0	47.2	52.6	36.2	16.4	52.8	91	424
	10	T	2	18.5	20.4	16.6	3.8	65.4	71.8	59.3	12.5	34.6	10	0
	6	T	3	29.3	31.3	26.6	4.7	43.6	51.1	36.7	14.4	56.4	14	0
White pine	4	U	1	24.6	25.2	24.0	1.2	63.2	65.2	60.7	4.5	36.8	52	6
	8	U	2	41.3	42.7	39.7	3.0	39.3	41.7	34.4	7.3	60.7	74	76
	8	U	3	73.1	75.8	69.7	6.1	0.0	0.0	0.0	0.0	100.0	147	191
	7	T	1	15.9	18.4	13.4	5.0	72.8	76.5	68.5	8.0	23.2	6	0
	10	T	2	23.5	25.8	21.4	4.4	54.7	58.7	50.4	8.3	45.3	10	0
	10	T	3	38.1	41.1	33.7	7.4	23.9	34.4	15.4	19.0	76.1	15	0
Yellow pine	10	U	2	46.1	51.7	40.8	10.9	23.7	42.7	0.0	42.7	76.3	312	192
	10	T	2	21.2	22.9	20.2	2.7	56.2	62.4	50.7	11.7	43.8	7	0
Poplar	8	U	1	41.7	49.4	34.7	14.7	44.5	51.6	37.3	14.3	55.5	325	385
	6	U	2	56.6	68.3	51.5	16.8	16.5	22.2	0.0	22.2	83.5	339	453
	4	U	3	86.3	87.3	84.0	3.3	0.0	0.0	0.0	0.0	100.0	330	300[b]
	8	T	1	16.6	18.3	14.5	3.8	65.9	70.7	63.1	7.6	34.1	7	0
	10	T	2	24.9	27.6	23.0	4.6	50.5	55.1	45.3	9.8	49.5	10	0
	8	T	3	35.8	41.6	30.9	10.7	31.3	39.1	25.8	13.3	68.7	9	0
Red wood	4	U	2	41.1	42.7	38.5	4.2	40.0	42.1	38.0	4.1	60.0	62	2
	6	T	2	16.5	19.6	15.4	4.2	63.9	72.3	58.8	13.5	36.1	1	0

[a] T = treated; U = untreated.
[b] Duration of glow was not recorded after 5 min.

At the end of the specified time, the specimen was removed from the furnace and placed on a fire brick with the exposed (burned) face of the specimen up. The length of time the specimen continued to flame or glow was noted and recorded. After cooling to room temperature the specimen was again weighed and the loss in weight calculated. The specimens were then photographed, the char was scraped off, and they were cut at the point of smallest cross-section. An imprint of the end of the specimen was made by use of carbon paper and the unburned area measured by means of a planimeter.

The temperature of the furnace was measured by means of an 8-gage iron constantan thermocouple. The tip of the thermocouple was placed in the top of the furnace immediately below the line of contact of the two specimens and extended 3 in. over the furnace. The furnace was heated until the temperature reached 1700 F. ± 25 F. and kept constant at this temperature. In practically all cases the tests were begun when the registered temperature was at a maximum of 1720 F. The presence of the cold samples caused an immediate drop in temperature of from 80 to 180 F., the lowest point usually being reached in ½ min., after which the temperature rose gradually to 1680 F. or 1690 F. at the end of 2 min. and to 1720 F. at 2½ min. (that is ½ min. after removal of the specimens for treated samples).

TABLE XII.—SUMMARY OF DATA FROM TABLE XI FOR 2-MIN. EXPOSURE ONLY.

Kind of Wood	Number of Specimens Tested	Treatment[a]	Loss in Weight, per cent				Percentage of Area Unburned				Percentage of Area Burned	Flame, sec.	Glow, sec.
			Avg.	Max.	Min.	Diff.	Avg.	Max.	Min.	Diff.			
Ash	8	U	34.8	39.4	29.9	9.5	44.6	50.7	39.1	11.6	55.4	74	242
	10	T	17.6	20.0	15.6	4.4	64.4	68.7	61.7	6.8	35.6	9	0
Birch	6	U	40.7	46.8	37.0	9.8	36.6	50.8	8.9	41.9	63.4	372	236
	8	T	18.5	15.7	19.9	4.2	66.6	72.5	58.9	13.6	33.4	5	0
Chestnut	8	U	42.2	49.7	36.8	12.9	32.2	39.1	21.4	17.7	67.8	73	5
	10	T	22.4	23.4	19.8	3.6	55.1	60.5	51.3	9.2	44.9	4	0
Douglas fir	7	U	30.9	35.0	28.0	7.0	57.1	63.1	51.2	11.9	42.9	44	20
	8	T	18.9	21.4	17.3	4.1	66.1	68.5	64.0	4.5	33.9	9	0
Maple	8	U	53.2	75.1	44.4	30.7	21.4	38.2	0.0	38.2	78.6	409	864
	10	T	21.2	22.4	20.0	2.4	58.3	64.0	47.8	16.2	41.7	13	0
Red oak	6	U	39.4	41.3	36.7	4.6	46.5	55.0	40.3	14.7	53.5	251	83
	10	T	19.9	21.0	18.5	2.5	59.9	63.8	53.4	10.4	40.1	11	0
White oak	6	U	49.5	56.1	45.3	10.8	21.3	38.6	7.6	31.0	78.7	301	1784
	10	T	19.0	20.8	16.7	4.1	70.2	77.5	67.0	10.5	29.8	10	0
Walnut	8	U	35.4	40.7	32.7	8.0	47.2	52.6	36.2	16.4	52.8	91	424
	10	T	18.5	20.4	16.6	3.8	65.4	71.8	59.3	12.5	34.6	10	0
White pine	8	U	41.3	42.7	39.7	3.0	39.3	41.7	34.4	7.3	60.7	74	76
	10	T	23.5	25.8	21.4	4.4	54.7	58.7	50.4	8.3	45.3	10	0
Yellow pine	10	U	46.1	51.7	40.8	10.9	23.7	42.7	0.0	42.7	76.3	312	192
	10	T	21.2	22.9	20.2	2.7	56.2	62.4	50.7	11.7	43.8	7	0
Poplar	6	U	56.6	68.3	51.5	16.8	16.5	22.2	0.0	22.2	83.5	339	453
	10	T	24.9	27.6	23.0	4.6	50.5	55.1	45.3	9.8	49.5	10	0
Redwood	4	U	41.1	42.7	38.5	4.2	40.0	42.1	38.0	4.1	60.0	62	2
	6	T	16.5	19.6	15.4	4.2	63.9	72.3	58.8	13.5	36.1	1	0

[a] T = treated; U = untreated.

The results obtained in these tests are given in Tables XI to XIV.

The great majority of these tests were carried out in series of eight or ten specimens but only the averages are given here.

Tests on Built-Up Flooring:

In applying the timber test to built-up flooring containing fire-retardant core and back and untreated face, it is found impractical to use the unburned area, or continuation of flame or glow as measures of its fire-resistive qualities because the untreated face veneer of $\frac{3}{16}$ in. thickness usually separates from the core due to failure of the glue under the influence of the heat. Furthermore, in removing the char prior to cutting the sample at its smallest cross-section, the face veneer separates from the core and flakes off. As a result the unburned area cannot be determined accurately. It is observed also that the untreated material continues to flame or glow after removal from the furnace for varying periods. However, an excellent measure of the degree of fire resistance can be obtained from a determination of the percentage loss in weight. That this is possible is shown by the data given in Table XIII in which is shown the average results. The natural resistance of hardwoods to fire penetration is supported by the fire-retardant core and backing to a marked extent.

Table XIII.—Tests on Built-Up Flooring.

Containing $\frac{3}{16}$ in. untreated face, $\frac{15}{32}$ in. treated chestnut core and $\frac{3}{16}$ in. treated chestnut backing. Tested at 1700 F for 2 min. Untreated face exposed.

Face Material	Number of Tests	Loss in Weight, per cent				Average Flame, sec.	Average Glow, sec.
		Avg.	Max.	Min.	Diff.		
Red oak	8	21.3	22.2	20.3	1.9	27	0
White oak	8	24.4	25.2	19.8	5.4	50	13
Teak	10	20.5	23.2	18.5	4.7	60	4
Walnut	8	21.6	23.7	19.7	4.0	53	77

Table XIV.—Tests on Built-Up Flooring Exposing the Fire-Retardant Backing to the Furnace at 1700 F. for 2 min.

Sample	Face Material	Loss in Weight, per cent	Flame, sec.	Glow, sec.	Oak Face	
					Flame, sec.	Glow, sec.
No. 1	Chestnut backing exposed, white oak face unexposed	18.5	0	0	77	22
No. 2		17.7	0	0	67	11
No. 3		18.8	0	0	52	8
No. 4		20.9	10	0	37	164
No. 5		18.5	0	0	58	8
No. 6		18.0	0	0	54	3
No. 7		18.3	0	0	12	0
No. 8		20.6	0	0	65	0
Average 8 samples		18.9	1	0	53	27

Comparison of the data in Table XIII with that in Table XII indicates the value of the fire-retardant core stock in preventing a great loss in weight. In other words, the loss in weight for the built-up flooring is slightly greater than that for completely treated material but is much less than that for untreated material. The duration of flame and glow is also decreased markedly.

In testing built-up flooring, one series of samples containing untreated white oak face was tested with the fire-retardant chestnut backing exposed to the furnace. The lower losses in weight obtained indicate clearly the increased fire resistance of the treated material. They further show the tendency of the unexposed untreated white oak face to continue to flame and glow. These data are given in Table XIV.

Examination of these tables discloses the following:

1. Of all untreated specimens (127) of both hard and soft woods tested at 2 min., only one specimen (red oak) retained sufficient unburned area to pass the New York Department of Buildings requirements (45 per cent for soft woods and 55 per cent for hard woods) and all failed to meet the flame and glow requirements.

2. Of all treated specimens (169) tested at 2 min., three chestnut, two maple and one red oak failed to meet the requirements as to unburned area, and one fir (17 sec.), two maple (16 sec.) and one red oak (16 sec.) failed to meet the requirements as to duration of flame (15 sec.) by a margin of 1 to 2 sec.

3. The percentage loss in weight and per cent of area burned per minute of exposure is greater during the first minute than in succeeding minute intervals. This shows the effect of the charcoal in retarding the combustion, which is appreciable even in such comparatively thin material ($\frac{3}{4}$ in.). It is evident that the rate of loss in both weight and area with time increases more rapidly for untreated than for treated wood. This shows the effect of the fire-retardant chemicals in forming a binder for the charcoal, causing it to become clinker-like and thus prevent fire penetration.

4. The percentage loss in weight for a 2-min. exposure for treated material is roughly about one-half that for untreated material, while the per cent of area burned is approximately three fourths.

5. The percentage loss in weight is approximately one half as great as the per cent of area burned for treated material and two thirds as great for untreated material.

6. The difference between maximum and minimum values for percentage loss in weight in the different groups is usually much less than the corresponding difference for unburned area except of course where the samples burned through. The average of these differences for percentage loss in weight is 4.2 for treated and 8.0 for untreated and for per cent of area unburned 11.5 for treated and 19.0 for untreated, showing that the values for percentage loss in weight are more nearly uniform.

7. The maximum time of exposure is limited to 3 min. in the case of practically all untreated woods because the specimens are burned through completely. This is very evident in the case of chestnut, pine and poplar and would probably be true of maple also.

8. The percentage loss in weight does not vary directly with the per cent of area burned in all cases. This shows the effect of natural variations in the texture or structure of the wood such as hardness, worm holes, etc., and indicates clearly the advantage of using the percentage loss in weight as a measure of fire-retardant values instead of the per cent unburned area. The protective influence of the char is also indicated here. The widest variation in per cent unburned area is noticeable in chestnut where worm holes were prevalent and in untreated maple and white oak where a pronounced tendency to glow is evidenced.

9. The height of blaze above treated samples during test is always of the same order of magnitude as that of the unobstructed furnace flame (that is, about 6 in.) but in case of untreated wood is usually from 12 to 24 in.

10. The time required for making the timber test after having prepared the samples is much greater when using unburned area as a means of measurement than when using loss in weight. This was shown clearly in one series of 70 samples where the time required for each operation was as follows:

(*a*) *Determining Unburned Area:*

1. Preliminary determination of dimensions	25 min.
2. Removing char, cutting, printing	5 hr.
3. Measuring unburned area	3 hr.

Total time 8 hr., 25 min. or an average of 7.2 min. per sample.

(*b*) *Determining Loss in Weight:*

1. Preliminary weighing	32 min.
2. Final weighing	40 min.

Total time 72 min. or an average of 1.03 min. per sample.

This shows a saving of time of more than 6 min. per sample in favor of the determination of loss in weight. It is therefore a decided step toward simplification.

11. The use of a specimen 10 in. long and a furnace 6 in. in diameter gives an exposure of 60 per cent of the lower surface of the specimen to the fire. Since the walls of the furnace are at a full red heat an additional burning effect is obtained in this connection which amounts to an additional 20 per cent. In the same manner a burning effect is obtained from the flanking bricks which are also at a red heat. Thus we have an exposure of approximately 50 per cent of the surface area of the specimen to the furnace. The effect of the flanking brick

upon the specimen is particularly noticeable when testing untreated material, the upper surface of such specimens invariably burning to a marked extent.

12. The time of exposure of the specimens in this series of tests has been varied from 1 to 4-min. Untreated wood decomposes to such an extent at 4-min. exposure that it is impossible to handle the specimens afterwards satisfactorily and in many cases the specimens fall apart and into the furnace. Consequently for comparative purposes an exposure of 3 min. was taken as the maximum and the data for 4 min. omitted. While all calculations were made from data obtained in an exposure of 2 min., it is likely that an exposure of 3 min. is preferable since specimens will withstand this longer exposure.

13. The importance of the information on duration of flame and glow is shown for both treated and untreated wood. While there is very little relation between the time of exposure and the duration of flame or glow, there is a qualitative relation between duration of flame and glow and the presence of untreated material. There is also a relation between the duration of glow and percentage loss in weight and percentage of unburned area as is shown particularly in specimens of untreated maple, red oak and white oak. Both maple and white oak show a longer duration of glow and a greater loss in weight and area burned than red oak. This natural tendency of the wood to disintegrate through smoldering must be overcome by the fire-retardant treatment in order to place these woods upon a par with red oak or chestnut which shows less tendency to glow.

14. In making measurements by means of a planimeter, extreme care is necessary to get readings of an accuracy of less than 0.02 in. With areas of the order of magnitude involved here this would amount to about 2 per cent. We would consider then that this value represents the accuracy with which such planimeter readings would ordinarily be made.

15. In all cases the charred portion of untreated woods was softer and more easily scraped off than that on treated wood due to the binding or clinkering effect of the chemicals upon the latter.

Conclusions (*Timber Test*):

The timber test offers a suitable method for measuring the combustibility of fire-retardant wood and is particularly valuable in giving information on percentage loss in weight or strength, resistance to flame penetration and tendency to spread fire as shown by continuation of flame and tendency to smolder or glow.

The use of the percentage loss in weight is considered more desirable as a measure of fire-retardant value than the use of percentage unburned area due to the greater uniformity of results and also to the saving in time.

A 10-in. specimen is preferable to one 12 in. in length.

VENEER AND BUILT-UP WOODWORK

The construction of veneered material is such as to permit a study of the effect of the presence of varying amounts of treated and untreated material upon its combustibility. This investigation was undertaken to determine the effect of treated and untreated crossbanding and face veneers upon the fire resistance of panels constructed of fire-retardant wood. The results given indicate clearly the effect of treated and untreated crossbanding upon the fire resistance of veneered stock.

A number of tests used heretofore and some variations were investigated with the result that the crib test and the fire-tube test were found to give the most satisfactory results. In this investigation the crib test has been extended so as to include percentage loss in weight of the test specimens as a measure of combustion loss or fire resistance and this factor alone offers a very satisfactory and convenient method of testing veneers containing fire-retardant crossbanding.

The material used consisted of samples of 5-ply veneer panels, varying in thickness from $\frac{1}{2}$ in. to $1\frac{1}{32}$ in. Each panel contained a core of chestnut or poplar, crossbanding of $\frac{1}{8}$ or $\frac{1}{12}$-in. poplar, face veneer of $\frac{1}{28}$-in. untreated walnut and back veneer of $\frac{1}{28}$-in. poplar, all glued with hide glue. In order to obtain more complete and comparable data some panels containing untreated core stock were included in these tests. The thickness and treatment of the crossbanding and core were as follows:

Sample	Core[a]	Crossbanding[a]	Face[a]	Back[a]	Total Thickness, in.
A	$\frac{23}{32}$-in. chestnut—T	$\frac{1}{8}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$1\frac{1}{32}$
B	$\frac{23}{32}$-in. chestnut—T	$\frac{1}{8}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$1\frac{1}{32}$
C	$\frac{23}{32}$-in. chestnut—T	$\frac{1}{12}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{15}{16}$
D	$\frac{23}{32}$-in. chestnut—T	$\frac{1}{12}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{15}{16}$
E	$\frac{23}{32}$-in. chestnut—T	$\frac{1}{12}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—T	$\frac{15}{16}$
F	$\frac{5}{16}$-in. poplar—T	$\frac{1}{8}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{5}{8}$
G	$\frac{5}{16}$-in. poplar—T	$\frac{1}{8}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—T	$\frac{5}{8}$
H	$\frac{5}{8}$-in. chestnut—U	$\frac{1}{12}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{7}{8}$
J	$\frac{5}{8}$-in. chestnut—U	$\frac{1}{12}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{27}{32}$
K	$\frac{1}{4}$-in. poplar—U	$\frac{1}{12}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{1}{2}$
L	$\frac{1}{4}$-in. poplar—T	$\frac{1}{12}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{15}{32}$
M	$\frac{1}{4}$-in. poplar—U	$\frac{1}{12}$-in. poplar—U	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{15}{32}$
N	$\frac{1}{4}$-in. poplar—T	$\frac{1}{12}$-in. poplar—T	$\frac{1}{28}$-in. walnut—U	$\frac{1}{28}$-in. poplar—U	$\frac{15}{32}$

[a] T = treated; U = untreated.

Samples were submitted to tests as follows:

1. *Torch Test:*

A gasoline blow torch has been used by previous investigators for making tests similar to those described here; consequently experiments with such a torch were made. It was found, however, that the length of the flame and its temperature varied directly with the amount of pressure within the torch, and to such an extent as to make the test unsuitable. Consequently, in order to have more constant and controllable conditions a bunsen burner flame was found preferable.

TABLE XV.—RESULTS OF FURNACE TEST.

Sample	Thickness, in.	Core, in.[a]	Crossband[a]	Face[a]	Time of Exposure, min.	Crossband		Face	
						Flame, sec.	Glow, sec.	Flame, sec.	Glow, sec.
A	1	$\frac{3}{4}$ T	U	U	2	72	390	Consumed	
B	1	$\frac{3}{4}$ T	T	U	2	0	30	Consumed	
C	1	$\frac{3}{4}$ T	T	U	2	0	14	Consumed	
D	1	$\frac{3}{4}$ T	U	U	2	24	280	Consumed	
E	1	$\frac{3}{4}$ T	T	U	2	0	0	4	35
F	$\frac{5}{8}$	$\frac{5}{16}$ T	U	U	2	112	322	Consumed	
G	$\frac{5}{8}$	$\frac{5}{16}$ T	T	U	2	5	15	Consumed	
No. 1	1	$\frac{5}{8}$ T	T	U	1	0	0	0	35
No. 1	1	$\frac{5}{8}$ T	T	U	2	9	0	0	60
No. 2	1	$\frac{5}{8}$ T	U	U	1	36	0	0	212
No. 2	1	$\frac{5}{8}$ T	U	U	2	0[b]	0	0	85
No. 3	$\frac{1}{2}$	$\frac{5}{16}$ T	T	U	1	0	0	0	57
No. 3	$\frac{1}{2}$	$\frac{5}{16}$ T	T	U	1	3	0	0	56
No. 4	$\frac{1}{2}$	$\frac{5}{16}$ T	U	U	1	32	0	0	133
No. 4	$\frac{1}{2}$	$\frac{5}{16}$ T	U	U	1	44	0	0	146

[a] T = treated; U = untreated.

[b] Crossband apparently burned completely in 2-min. exposure, hence no flame for untreated. One-minute exposure seemed to give more complete data.

2. *Furnace Test:*

(*a*) *Exposing Flat Surface of Panel.*[1]—The sample (6 by 12 in.) was placed flat upon two fire bricks 3 in. apart over a 6-in. gas crucible furnace with the walnut face exposed to the fire at a temperature of 1700 F. for 1 and 2 min. Results of the tests are shown in Table XV. There was no noticeable difference in appearance between treated and untreated crossbands. There was a marked difference in the length of time during which flame and glow continued.

(*b*) *Exposing Edge of Panel.*—This suggested itself as a possible method of determining the effect of heat upon a separation of the layers of veneer, but proved inconclusive. The sample was placed on edge for 1 min. instead of flat as explained above. The flame and glow record shows a slight difference. The results are given below:

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[1] This test was suggested by Mr. George Strehan, Consulting Engineer, New York City, in a manuscript under date of December, 1912, as "Slab Test." Mr. Strehan specified no time requirement for the application of heat.

SAMPLE	FLAME, SEC.	GLOW, SEC.
A—Untreated crossband	15	55
B—Treated crossband	3	0
C—Treated	0	8
D—Untreated	3	0
E—Treated	2	0
F—Untreated	4	85
G—Treated	0	0

Visible evidence shows very little difference. Flame and glow results are inconclusive.

3. *Horizontal-Flame Test:*

A similar test has been described by H. A. Gardner,[1] as a torch test. A 3-in. blue bunsen flame was used with a measured temperature of approximately 1200 F. at the tip. The sample was placed on edge in a vertical position and was exposed for 2 min. to the flame of a bunsen burner placed on its side so that the flame projected horizontally, the open burner tip being ½ in. from the sample. The flame and glow record follows:

SAMPLE	FLAME, SEC.	GLOW, SEC.
A—Untreated	50	65
B—Treated	0	0
C—Treated	0	0
D—Untreated	4	125
E—Treated	0	0
F—Untreated	3	213
G—Treated	0	0

Visible differences were not sufficiently pronounced to be noticeable. There was a marked difference in duration of flame and glow.

4. *Flame-Penetration Test:*

Using a 3-in. blue flame as in the horizontal-flame test referred to above, the sample was placed flat on an iron ring 9 in. high. A ¼-in. asbestos board, having a hole 3 in. in diameter, was placed beneath the sample and the exposure was continued for 1 and 2 min. on 12 samples. These tests gave no data on flame and glow, and flame penetration was from $\frac{3}{64}$ to $\frac{3}{32}$ in. for treated and untreated samples. On cooling, the crossbanding usually separated from the treated core stock and this made the determination of flame penetration an unreliable indicator. The charred shell formed by the crossbanding served to insulate and protect the wood from the flame and so reduced the extent of burning. It is probable that this seemingly desirable test is not applicable as a rapid test of built-up woodwork or veneer stock.

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[1] *Bulletin 42*, Educational Bureau, Paint Manufacturers Assn.; see also *Lumber World Review*, February 10, 1914, p. 20.

5. *45-deg.-Angle Test:*

This test was adapted from the Gardner data but it failed to give information on the tendency of veneers to separate under heat and the tendency of the veneers to burn.

(*a*) A 3-in. blue flame was used as in the horizontal flame test referred to above. The sample was clamped at one end and held at an angle of 45 deg. The lower edge was exposed (2 in. above the burner) to the flame for 1 min. The results obtained in this test are given below:

Sample	Crossband Treatment[a]	Face Material and Treatment[a]	Face		Back Material and Treatment[a]	Back	
			Flame, sec.	Glow, sec.		Flame, sec.	Glow, sec.
A	U	Walnut—U	25	0	Poplar—U	292	0
B	T	Walnut—U	0	0	Poplar—U	5	0
C	T	Walnut—U	3	0	Poplar—U	0	0
D	U	Walnut—U	0	0	Poplar—U	353	0
E	T	Walnut—U	0	0	Poplar—U	0	0
F	U	Walnut—U	138	0	Poplar—U	120	0
G	T	Walnut—U	0	0	Poplar—U	0	0

[a] T = treated; U = untreated.

Some visible difference was noticeable but not sufficient to justify drawing definite conclusions. In all cases the burning of the face and back veneers introduced factors which gave a marked effect upon the results. The duration of flame record indicated a distinct difference between the treated and untreated material. A visible difference was also noticed between the effect of the walnut and poplar face and back veneers, which can probably be attributed to the difference in the density of these woods.

(*b*) The use of a 6-in. blue flame gave practically the same results as with the 3-in. flame. A more marked visible difference was noticeable on poplar back veneer side.

6. *Fire-Tube Test:*

The fire-tube test known as the Dunlap Fire-Tube Test[1] involves exposing the specimen for a period of 4 min. to an 11-in. ± ½ in. flame at a temperature approximately 1000 C. (1830 F.) so adjusted as to give a temperature of approximately 180 C. (355 F.) at the top of the tube. Schlegel[1] has used a modification of the fire-tube test in his studies on the fire-retarding action of chemicals. He used a specimen approximately $\frac{3}{8}$ by $\frac{3}{8}$ by 4 in. and a smaller igniting flame.

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[1] T. R. Truax and C. A. Harrison, "A New Test for Measuring the Fire Resistance of Wood," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 973 (1929).

It appeared likely that the height of the flame used in this test as well as the high temperature to which the specimen is exposed would give valuable information, particularly upon the separation of the veneer layers. Accordingly specimens were prepared from 7 panels, each specimen being $\frac{3}{8}$ in. thick, 40 in. long with a width corresponding to the thickness of the panel. For example, the specimens from sample A were cut $\frac{3}{8}$ by $1\frac{1}{32}$ by 40 in. instead of the usual size $\frac{3}{8}$ by $\frac{3}{4}$ by 40 in. used in the fire-tube test.

The average results of these tests are given in Table XVI.

This table shows that the fire-tube test gives a clear distinction between those panels which contain fire-retardant crossbanding and those containing untreated crossbanding. In the case of panels which contain fire-retardant crossbanding it is noticed that there is close correlation in the percentage loss in weight and the maximum temperature at the top of the tube. In addition, the thickness of the crossbanding seems to show its influence. For example, sample A containing $\frac{1}{8}$-in. poplar crossbanding showed a percentage loss in weight of 49 whereas sample D with $\frac{1}{12}$-in. poplar crossbanding showed a percentage loss of 43. The maximum temperature at the top of the tube was 1214 and 895 F., respectively.

TABLE XVI.—FIRE-TUBE TESTS ON VENEERED STOCK. AVERAGE RESULTS.

Sample	Number of Tests	Treatment[a]				Percentage of Untreated Material	Loss in Weight, per cent				Temperature at Top of Tube, deg. Fahr.			
		Core	Crossband	Face	Back		Avg.	Max.	Min.	Diff.	Avg.	Max.	Min.	Diff.
A	7	T	U	U	U	30.9	49	62	37	25	1214	1350	825	525
B	8	T	T	U	U	6.9	21	26	17	9	341	385	310	75
C	9	T	T	U	U	8.2	23	25	20	5	361	420	300	120
D	7	T	U	U	U	17.9	43	56	31	25	895	1275	470	805
E	5	T	T	U	T	4.1	22	25	21	4	343	410	310	100
F	5	T	U	U	U	37.6	48	54	45	9	1305	1320	1285	35
G	5	T	T	U	T	7.0	18	19	16	3	360	375	340	35

[a] T = treated; U = untreated.

With respect to the separation of the veneer layers due to the breaking down of the glue, it was found that separation took place only in those specimens containing untreated crossbanding. This was apparently due to the extensive burning of the sample caused by the presence of the untreated crossbanding.

It should also be pointed out that in no case where the sample contained fire-retardant crossbanding was there any extensive burning of the face or back veneers above the area of general burning covered by the igniting flame. This might be attributed to the quenching influence of the treated material on the spread of flame in the untreated material.

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[1] R. Schlegel, "Untersuchungen über die Grundlagen des Feuerschutzes von Holtz," Verlag Chemie, Berlin (1934).

Finally it should be noted that in all cases when the specimen contained untreated crossbanding a blaze of 6 to 18 in. in height appeared above the top of the tube. When, however, the specimen contained fire-retardant crossbanding the blazing of the specimen was confined to the lower half of the specimen.

It would appear that the fire tube offers a possible method for testing veneer stock. The outstanding disadvantage of this test is the fact that pronounced warping of the specimen often takes place necessitating the operator constantly following the lower end of the specimen with the flame. This introduces the personal equation of the operator, which is undesirable. This warping acts also as a damper upon the flame, resulting in marked variations in the temperature at the top of the tube. As a result a direct relationship between loss in weight and built up temperatures, as shown at the top of the tube, is not always obtained. This is shown by comparison of samples B and G or C and G in the averages of Table XVI. Furthermore, the fact that the test specimen must be 40 in. in length offers a practical handicap in obtaining samples for testing. If shorter pieces are obtained and spliced together so as to obtain a 40-in. sample, the test is still more unsatisfactory due to more extensive warping and requiring special care in preparing the test specimens.

The fire-tube test does not show a uniform relation between the amount of untreated material present, the total loss in weight and the temperature at the top of the tube. This is shown clearly in comparing samples A, D and F. Sample A containing almost 31 per cent of untreated material shows a loss in weight of 49 per cent and a temperature of 1214 F., while sample D, containing practically 18 per cent of untreated material, shows almost the same loss in weight but much lower temperature (895 F.), and sample F, containing nearly 38 per cent of untreated material shows very little more loss in weight than sample D and even less than sample A, which has less untreated material present.

It is also noted that quite large differences between maximum and minimum values are obtained both in temperature and weight loss measurements, particularly in cases where untreated material is present. The presence of fire-retardant material reduces these extremes considerably.

7. *Crib Test:*

In determining the fire-resistant value of the material used in these tests four factors have been used as a basis of comparison. These are the height of the blaze formed by the burning crib, the duration or continuation of flame or blaze after removal of the igniting flame, the duration of glow, and the percentage loss in weight.

TABLE XVII.—COMPARATIVE RESULTS OBTAINED USING EIGHT AND TWELVE STICKS PER CRIB, 1 MIN. EXPOSURE, 1200 F.

Averages of six tests.

NOTE.—The pieces were numbered as follows: Bottom layer from front to back Nos. 1, 2, 3, 4; second layer from left to right Nos. 5, 6, 7, 8; third layer from front to back Nos. 9, 10, 11, 12.

Sample	Treatment[a]		Percentage of Untreated Material	Number of Pieces in Crib	Average Loss in Weight, per cent	Average Flame, sec.	Average Glow, sec.	Blaze Height from Burning Crib, in.
	Core	Cross-band						
A	T	U	39.3	8	13.8	142	180	6
				12	17.7	274	320	10
B	T	T	14.3	8	4.8	11	4	4
				12	5.5	25	0	4
C	T	T	14.3	8	5.1	17	15	4
				12	5.7	20	0	4
D	T	U	30.9	8	9.5	65	201	12
				12	10.1	110	120	8
E	T	T	7.1	8	4.7	5	18	4
				12	4.8	8	22	4
F	T	U	10.7	8	12.9	195	250	6
				12	57.4	653	741	10
G	T	T	7.1	8	4.2	0	3	4
				12	3.7	0	4	4
H	U	T	83.3	8	18.0	109	72	10
				12	78.8	346	460	14
J	U	U	100.	8	81.2	334	626	18
				12	87.3	275	552	24
K	U	T	65.9	8	6.1	29	19	4
				12	7.7	49	0	4
L	T	U	46.7	8	34.7	234	315	12
				12	68.4	526	693	16
M	U	U	100.	8	88.3	521	993	18
				12	92.0	325	1115	24
N	T	T	15.2	8	4.2	6	6	4
				12	4.3	0	4	4

[a] T = treated; U = untreated.

In cases where the sample had a thickness of more than $\frac{5}{8}$ in., specimens were cut to the regulation size of $\frac{1}{2}$ by $\frac{1}{2}$ by 6 in. before testing. The samples were prepared so that one-half the sticks had the poplar back veneer side left, and one-half had the walnut face veneer left. In other cases, where the thickness was $\frac{5}{8}$ in. or less, the samples were cut to $\frac{1}{2}$ in. by that thickness, thus having both back and face veneers present. Using 8 and 12 pieces for each test, the samples were arranged in crib form so that all veneer sides (face or back) were turned toward the center. In other words, the center opening had veneer exposed on all four sides. The bottom layer (and alternate layers) was composed of the sticks having the walnut face veneer left and the second layer (and alternate layers) of the sticks having the poplar back veneer left. Six tests upon each sample were made using 8 pieces per crib and usually one test using 12 pieces per crib for check purposes. The average results obtained in these tests are given in Table XVII.

In view of the fact that the results obtained in exposures of 1 min. do not give a correct measure of the amount of untreated material present, a series of tests was made on certain samples using 12 pieces per crib, and testing at a temperature of 1200 F. for 2 min. It was found that the percentage loss in weight corresponded more closely to the actual amount of untreated material present than when tested for 1 min. The presence of untreated material protected on two sides by treated crossbanding was not shown in 1-min. tests. However, the 2-min. exposure showed this up clearly. This is brought out particularly in sample K. The average results obtained in these tests, using 6 tests for each sample, are given in Table XVIII.

TABLE XVIII.—AVERAGE RESULTS FROM EXPOSURE OF CRIB OF TWELVE PIECES EACH TO A FLAME OF 1200 F. FOR 2 MIN.

Sample	Treatment[a]				Loss in Weight, per cent	Per-centage of Untreated Material	Blaze Height, in.	Flame, sec.	Glow, sec.	Number of Pieces Burning
	Core	Cross-band	Face	Back						
A	T	U	U	U	36.3	39.3	15	190	31	12
B	T	T	U	U	7.47	14.3	3	35	2	2
C	T	T	U	U	6.70	14.3	4	24	3	2.5
D	T	U	U	U	23.3	30.9	9	225	0.5	8.5
K	U	T	U	U	77.9	65.86	20	387	821	12
L	T	U	U	U	77.0	46.7	19	421	315	12
N	T	T	U	U	6.93	15.2	5	36	7	3.3

[a] T = treated; U = untreated.

Conclusions (*Veneer and Built-Up Woodwork*):

Fire-retardant crossbanding gives pronounced support to the fire-resistive qualities and affords protection against spread of flame in built-up panels containing treated core stock. The effect of fire-retardant back veneer $\frac{1}{28}$ in. thick is also noticeable as is shown by comparison of samples C and E as well as sample G in Table XVII.

The crib test offers a suitable and adequate means of measuring the fire resistance of veneered panels; particularly, if the test is enlarged to include the percentage loss in weight of the samples, and the exposure is for a minimum of 2 min.

The presence of untreated crossbanding causes an increase in the duration of flame and glow, percentage loss in weight and height of blaze from the burning crib. It is also evident that the percentage loss in weight varies directly with the duration of flame and glow. This would seem to justify the use of the weight loss as a means of measuring fire resistance or combustibility.

The mean percentage loss in weight for all samples containing treated core and crossbanding is 4.8 and that for treated core and untreated crossbanding 38.1. The differences between panels containing treated and untreated crossbanding are thus clearly defined. The differences in loss in weight as well as in flame and glow are sufficiently pronounced to form a basis for specification requirements.

It is noticed that the number of pieces used in each test makes little difference in a 1-min. exposure when the core and crossband are both composed of treated stock. This is shown in the averages for 8 pieces and for 12 pieces in the cases of samples B, C, E, G and N, where the loss in weight is of the same order. Such is also the case regarding a completely untreated panel as shown by samples J and M. However, where either core or crossbanding is of untreated material the differences observed when using 8 or 12 sticks per crib are very pronounced.

The case of sample K is very interesting. Here the total thickness of the panel was approximately ½ in. and both face and back veneers as well as crossbands were present. The protection given the untreated core by the treated crossbanding gives excellent evidence of the value of treated crossbanding. Although the sample contained over 65 per cent untreated material, its presence was not indicated by a 1-min. exposure. A 2-min. exposure, however, is sufficient to give a fairly accurate measure of the amount present.

Comparison of samples K and H further accentuates this protective influence. Sample H also contained untreated core stock, but being a thick panel only one side or edge of each crib piece was protected by the treated crossbanding, leaving three sides exposed to the flame and as a result fire effects were more pronounced.

It was also noticed that there is a relationship between the separation of the crossband from the core and the percentage loss in weight. In cases of treated crossband there was practically no flame spread and only the two center pieces of the bottom layer showed any separation of the crossband and core.

In cases where pronounced burning of the crib took place, the pieces would tend to separate at the middle and thus cause the crib to fall apart. In order to make the final weighing, it was necessary to collect these fragments carefully. This was done by placing a 12-in. square piece of fire-retardant veneer under the crib after the removal of the igniting flame and this served to catch all falling fragments.

While all types of test show a decided difference between the fire-resistant qualities of veneer containing fire-retardant crossbanding as against that containing untreated crossbanding, it is apparent that the most useful information is obtainable from the crib test and the fire-tube test.

It should be borne in mind that the crib test involves an exposure of the material to a temperature of approximately 1200 F. for 1 or 2 min., while the fire-tube test involves an exposure at approximately 1830 F. for 4 min. The results shown in Table XVIII seem to indicate that an exposure of 2 min. instead of 1 min. in the crib test gives a clearer line of demarcation between treated and untreated material, also a more accurate measure of the amount of untreated material present. The crib test, however, seems to offer certain advantageous features over the fire-tube test. Among these may be mentioned:

1. The difference between the percentage loss in weight for treated core and crossbanding as against treated core and untreated crossbanding is much greater in the crib test than in the fire-tube test. The crib test thus shows a greater ratio and places a higher value upon lower combustible content.

2. The height of blaze can be observed and measured more accurately in the crib test than in the fire-tube test. This is partially due to the effect of the draft in the latter. In many instances where the face or back veneer as well as the crossbanding was blazing slightly it was clear that the blaze was extinguished by the draft in the tube. Since the extent of blazing gives a measure of the amount of combustible material present as well as the tendency to communicate flame, the crib test seems superior at this point.

3. Spread of flame from stick to stick in the crib test gives a more reliable measure of the tendency to communicate flame than can be obtained in the fire tube where only one stick is used.

4. Crib test specimens can be prepared from a sample 6 by 7 in. in surface area. This is a highly important factor to the inspector when selecting samples for test since waste material can be utilized. If the 40-in. fire-tube-test specimen is made up from such a short sample the test is much less satisfactory due to the difficulty in preparing the sample and to warping during test. No splicing is necessary in preparing crib-test specimens.

The average loss in weight for panels containing treated core and crossbanding is approximately 5 per cent for 1 min. and 7 per cent for

2-min. exposure (Tables XVII and XVIII). For panels with treated core stock and untreated crossbanding the average loss in weight is 18 per cent using 8 pieces per crib and 38 per cent using 12 pieces per crib for 1-min. exposure and 77 per cent, using 12 pieces, for 2-min. exposure; for panels with untreated core and treated crossbanding 19 per cent using 8 pieces per crib and 79 per cent using 12 pieces for 1-min. exposure; and for panels completely untreated 85 per cent using 8 pieces and 90 per cent using 12 pieces per crib for 1-min. and 94 per cent for 2-min. exposure. The fact that a crib containing 12 pieces gives a greater percentage loss ratio between panels containing treated and untreated crossband is sufficient reason to justify using 12 pieces instead of 8. This is shown by Table XVII.

The average duration of flame and glow (using 12 pieces per crib) for panels containing treated core and crossbanding was less than 10 sec. for 1-min. exposure; for panels with treated core and untreated crossbanding—flame 393 sec., glow 481 sec.; for panels with untreated core and treated crossbanding—flame 132 sec., glow 140 sec.; and for untreated core and crossbanding—flame 364 sec., glow 572 sec.

The height of the blaze formed by the burning crib also gives a measure of the fire-retardant quality of the wood. Where both core and crossbanding were of fire-retardant material, the blaze height averaged less than 4 in., whereas in cases where either core or crossbanding was untreated, the height of the blaze was much greater.

In certain cases (samples J and M of Table XVII) it is observed that the duration of flame is less for cribs containing 12 pieces than for cribs containing 8 pieces. This is attributed to the fact that the sticks burned more rapidly in the former case due to the presence of more fuel or untreated wood.

The fire tube also offers a possible method for testing veneer material. Since the test specimen must have a length of 40 in. a greater quantity of material is required for test purposes unless the specimen is spliced. This causes even more warping of the specimen during testing than the already pronounced warping noticed in a single piece, and since the personal judgment of the operator is thus introduced it forms a disadvantage of this test. The construction of the fire tube makes it difficult to observe the height of the blaze during burning and impossible to observe the duration of glow, consequently the test is limited to a measure of loss in weight and temperature produced by the burning specimen.

The crib test has been extended to include a measure of the temperature built up above the burning crib and a direct relationship is found to exist between the percentage loss in weight and the temperature. This same relationship is evident in data obtained with the fire tube and it appears unnecessary to record two results whose meaning is synonymous.

SUMMARY

Of all tests studied, the crib, timber and fire-tube are the only ones which are considered suitable for testing fire-retardant wood. No single test is capable of giving all of the information desired. The crib test seems to be the best single test because it is capable of giving most information, requires a small amount of material for test specimens and requires no elaborate or expensive equipment for its performance. Sufficient information can be obtained from a combination of these tests to serve as a basis for specifications for fire-retardant treatments. The shavings test, although a desirable plant test because of the short time required to dry and test wet shavings, is unsuitable for testing kiln-dried material.

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The authors wish to express their thanks to Mr. Fred T. Perrine, New Jersey Veneer Co., and to Mr. J. H. Wagner, Central Veneer and Panel Co. for their kind cooperation in supplying the panels used in the veneer tests, also to Wood Mosaic Co., Lanham Hardwood Flooring Co., and Hasbrouck Flooring Co. for material used in the tests of built-up woodwork.

DISCUSSION

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Mr. C. R. Brown[3] (*by letter*).—The paper is a valuable contribution as a study of the shavings, crib, and timber tests. Some conclusions are based on tests with samples consisting of various mixtures of treated and untreated wood which would not be applicable to tests with samples made up of uniformly treated wood with various concentrations of treatment. In other tests, there is said to be a relation between the duration of flame and glow and the presence of untreated material. Although these can be said to indicate qualitatively the ature. This same relationship is evident in data obtained with the fire tube and it appears unnecessary to record two results whose meaning is synonymous.

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[1] Chemical Engineer and Chemist, Philadelphia, Pa.

[2] Director, Testing Laboratory, Protexol Corp., Kenilworth, N. J.

[3] Assistant Physicist, National Bureau of Standards, Washington, D. C.

SUMMARY

Of all tests studied, the crib, timber and fire-tube are the only ones which are considered suitable for testing fire-retardant wood. No single test is capable of giving all of the information desired. The crib test seems to be the best single test because it is capable of giving most information, requires a small amount of material for test specimens and requires no elaborate or expensive equipment for its performance. Sufficient information can be obtained from a combination of these tests to serve as a basis for specifications for fire-retardant treatments. The shavings test, although a desirable plant test because of the short time required to dry and test wet shavings, is unsuitable for testing kiln-dried material.

ACKNOWLEDGMENTS

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Loss in weight is concluded to be a direct measure of the amount of untreated material in the crib test with 2-min. exposure, considering the results with 65 per cent untreated material. While undoubtedly some relation exists, it is not uniform over the entire range as can be seen by comparing the data for various other amounts of untreated material. Also, loss in weight does not necessarily give an indication of the loss in strength, which may become appreciable before much loss in weight occurs, as has been shown by strength tests made at temperatures well below the ignition point. It is likewise doubtful if loss in weight or height of flame is a measure of the resistance to ignition. With regard to the conclusions made from the study of effect of moisture, while the reduction in weight loss is small in absolute figures, for the range 0 to 7 per cent moisture, it amounts to about 20 per cent relatively. Also, it is not certain that a uniform relation exists in the given moisture range.

In the discussion of the crib tests, mention is made of the relation between certain factors and the "fire-resistive qualities," but the latter term is not defined, nor is it stated how it is measured. In discussing the timber tests, reference is made to the "natural resistance of hardwoods to fire penetration." We do not know of any experimental proof of this assumption regarding hardwoods.

The shavings test is clearly not satisfactory for specification purposes, as shown by the paper, but it might be improved by using the percentage of shavings consumed as a criterion of failure rather than the duration of flaming. In the crib test, a larger number of tiers might be of value as it would permit a spread of flame beyond the region attacked by the burner flame. The timber test can hardly be expected to give any information on the resistance to flame penetration as the time of exposure is only 1 or 2 min.

The fire-tube tests were not made on solid specimens of standard size ($\frac{3}{4}$ in. wide), and therefore are not representative of the usual application of this method of testing. Consequently any conclusions based on them would be of limited value. It is not believed that the results for the specimens containing treated crossbanding vary sufficiently to establish a relation between loss in weight and maximum temperature. Too much significance should not be placed on the wider range in weight loss between treated and untreated wood afforded by the crib test in comparison with the fire-tube test as it is due in part to the difference in severity and duration of exposure to the burner flame in the two tests. We have found that reducing the time of exposure in the fire-tube test decreases the loss in weight of the treated specimens and thereby increases the range in weight loss between untreated and treated wood. It is also doubtful if it is an advantage to use waste material for tests because such material is not always representative.

MR. WILLIAMS (*by letter*).—A satisfactory test method should be capable of indicating the presence of untreated material in any part of the test specimen. In a practical application of tests to commercially treated material this is of great importance. The procedure here given wherein certain known amounts of untreated material are introduced into the test specimen appears to be a practical way of testing the test method. The very nature of the structure of wood prevents 100 per cent uniformity of treatment in commercial practice. A test method that is satisfactory where some untreated material is present would certainly be satisfactory for uniformly treated material.

We state that the relation between duration of flame and glow and the presence of untreated material is qualitative.

The data given show clearly that there is a direct relationship between loss in weight and amount of untreated material present.

In the tests as conducted here, particularly the timber test, the loss in weight indicates an actual destruction of the material, hence a loss in strength must follow. Further work on this particular point is in progress.

The conclusion with reference to moisture content is that for specification requirements a variation of the magnitude usually encountered in kiln-drying practice has no appreciable effect. In modern kiln-drying practice variations of more than 2 to 3 per cent are seldom encountered.

It has been shown in the work of Cherry[1] and Schwartz[2] that in general the resistance of wood to fire varies directly with the density.

Use has been made of the percentage of specimen burned in the shavings test as mentioned in the paper. However, the shavings test has the fundamental handicap of variable dimensions which cannot be overcome in kiln-dried wood.

From a practical standpoint no advantage can be gained by increasing the number of tiers in the crib test for a given igniting flame as shown in the results. It must be remembered that we are seeking a practical test.

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[1] R. T. Baker, "The Hardwoods of Australia" (1919).

[2] Ernest von Schwartz, "Handbuch zur Erkennung, Beurteilung und Verhütung der Feuer- und Explosionsgefahr," Munich (1922).

While a measure of flame penetration can probably be obtained in the timber test, the test is not recommended as such.

A large number of fire-tube tests have been made by one of us using specimens of the recommended dimensions $\frac{3}{8}$ by $\frac{3}{4}$ by 40 in., as a result of which it can be safely stated that: (1) the results are affected by warping, (2) no data are obtainable on continued flame or glow, (3) the length of specimen employed permits the presence of at least 50 per cent untreated material without any appreciable effect upon the results.

One series of tests using treated and untreated chestnut illustrates this fact clearly. Five specimens were entirely untreated, 5 entirely treated and 10 were spliced using one-half untreated and one-half treated material, 5 of which were tested with the treated end exposed to the flame and 5 with the untreated end exposed. The results were as follows:

	100-PER-CENT UNTREATED MATERIAL	100-PER-CENT TREATED MATERIAL	50-PER-CENT TREATED MATERIAL; TREATED END EXPOSED	50-PER-CENT TREATED MATERIAL; UNTREATED END EXPOSED
Percentage weight loss, max.	84	18	21	52
Temperature at top of tube, deg. Fahr., max.	1336	311	330	605

The fire-tube test in principle offers possibilities of development into a good practical test, but at present it is not a satisfactory test for commercially treated wood and the authors understand it has never been so recommended by its originators. As a result of experience gained in testing commercially treated wood it is felt that the fire tube in its present state of development does not properly detect or give a measure of the presence of untreated or streaky material.

Veneered woodwork is universally employed for interior woodwork. Much of this material is manufactured from fire-retardant wood. The authors feel that they have been justified in attempting to make use of the fire tube for testing such material. If, as Mr. Brown suggests, the test is subject to error as a result of slight variations in the dimensions of the specimen, this would only serve to show it to be impractical. However, the authors have not found it to be subject to such weakness. Wide variations do exist between the temperature developed by the burning specimen even using uniformly treated material.

We are aware of the effect of time of exposure upon the results when comparing treated and untreated material. However, for a shorter exposure and at a lower temperature the crib test shows a wider variation than the fire-tube as between treated and untreated material due to the type of test specimen employed.

MESSRS. E. F. HARTMAN,[1] A. S. WILLIAMS[1] and R. C. BASTRESS[1] (*authors' closure by letter*).—The authors would like particularly to emphasize that the work described here was carried out for the purpose of developing test methods which are capable of being applied in a practical way to commercially treated fire-retardant wood. From a strictly scientific viewpoint certain other data such as density of material, kind and concentration of salts, etc., might rightly have been included. However, it is felt that the nature of the problem did not necessitate the inclusion of such data. A distinction is made between a test that is satisfactory in comparing uniformly treated material with different concentrations or kinds of chemicals on the one hand and a test capable of detecting the presence of untreated material or of varying degrees of treatment and so classifying them. It is the latter type of test in which the authors have been interested.

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[1] President; Director, Testing Laboratory; and Chief Engineer; respectively, Protexol Corp., Kenilworth, N. J.

THE DETERMINATION OF TRACES OF DISSOLVED OXYGEN BY THE WINKLER METHOD[1]

By M. C. Schwartz[2] and W. B. Gurney[3]

Introduction

This paper does not pretend to record a comprehensive investigation of the Winkler method of determining dissolved oxygen. The present discussion gives data and conclusions from an attempt to evaluate the performance of a deaerator.

The Winkler method has been the normally accepted procedure for the determination of dissolved oxygen in water in the industrial as well as the sanitary field. Modifications, such as the Rideal-Stewart, which have been introduced to compensate for the presence of various interfering substances, have made possible the measurement of dissolved oxygen within limits of error very small in comparison with the saturation values normal to atmospheric temperatures. Dissolved oxygen values of interest in estimating the performance of a modern power plant deaerator, however, are approximately of the order of these small limits of error. The bi-iodate modification which is offered as the principal contribution of this paper was developed as the result of an attempt to overcome the sensitivity limitations inherent in the normal procedure and provide simple evaluation of sources of error commonly encountered. It is believed that adoption of the procedure outlined will largely avoid the uncertainties which in the past have been associated with the Winkler method for determination of dissolved oxygen.

Precision of Method

The Winkler method of determining dissolved oxygen may be divided into two steps: (*A*) in which an amount of iodine is produced equivalent to the dissolved oxygen originally present, and (*B*) the determination of the iodine so produced. Since the concentration of iodine being determined is extremely low, a careful survey of the iodine-thiosulfate titration[1] as well as of such considerations[2] as are necessary for precise laboratory work is in order.

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[1] The present paper includes a portion of a program of boiler feedwater research being carried out at the Louisiana Steam Generating Corp. This work is being done under the general direction of Mr. E. B. Powell, Consulting Engineer of Stone & Webster Engineering Corp., Boston, Mass., with the cooperation of the Institute of Industrial Research, Louisiana State University, and with the collaboration of Mr. S. T. Powell, Consulting Chemical Engineer, Baltimore, Md.

[2] Research Chemist, Louisiana Steam Generating Corp., Baton Rouge, La.; and Boiler Feedwater Fellow, Institute of Industrial Research, Louisiana State University, Baton Rouge, La.

[3] Efficiency Engineer, Louisiana Steam Generating Corp., Baton Rouge, La.

To obtain an accurate evaluation of dissolved oxygen, the following points must be considered:

1. The sensitivity of the end point of the reaction is a function of the kind of starch used and the temperature at which the titration is carried out.
2. The apparent oxygen value obtained may be in error by using Winkler reagents which have been subjected to some unsuspected contamination.
3. The Winkler reagents contain dissolved oxygen.

To enable us to determine the true value of the dissolved oxygen or iodine equivalent in the prepared sample, irrespective of the source of the iodine present, we have modified the Winkler procedure slightly. The principles involved are not new, but their adaptation to the Winkler method of determining dissolved oxygen we believe is. The method consists in adding a small known amount of iodine from a standard potassium bi-iodate solution to two samples, one of which is a blank. Sensitivity of the test procedure is, of course, of great importance in the determination of traces of oxygen. With the procedure described, however, actual consideration of sensitivity becomes unnecessary, for the reason that the two determinations have a common sensitivity (cancelled in the calculation) since the same starch solution is used and the direction of reactions is the same in both. The principle of adding a definite quantity of constituent so that the sensitivity correction is relatively diminished in magnitude is one capable of general application and might well be used more generally in the analysis of very dilute solutions.

REAGENTS AND PROCEDURE FOR DETERMINING DISSOLVED OXYGEN

The following reagents were used:

Solution No. 1.—Manganese chloride: 425 g. per liter

Solution No. 2.—Alkaline potassium iodide: 700 g. KOH and 150 g. KI per liter

Solutions Nos. 3 and 4.—Sulfuric acid: concentrated acid and 1:1

Solution No. 5.—Approximately 0.1 *N* sodium thiosulfate: 24.8 g. per liter

Solution No. 6.—Potassium bi-iodate: 3.2499 g. per liter

Solutions Nos. 7 and 8.—Starch solutions: Arrowroot and Baker's soluble

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[1] I. M. Kolthoff and N. H. Furman, "Volumetric Analysis," Vol. II, Chapter 12, John Wiley and Sons, New York (1929).

[2] H. A. Fales, "Inorganic Quantitative Analysis," Chapter 4, The Century Co., New York (1925).

The collection of a proper sample for the determination of dissolved oxygen requires considerable care. Two samples are collected in series in pyrex Erlenmeyer flasks, one sample to be used as a blank, the other for the dissolved oxygen determination. The blank is treated with reagents in the order, No. 2, No. 3, and No. 1, whereby the effect of the presence of the Winkler reagents is secured without fixing any of the oxygen dissolved in the sample. The sample on which the oxygen determination is to be made is treated with the reagents Nos. 1, 2, and 3 in their numerical order. It is very important that the temperature of the solution to be tested should not be above 70 F.[1,2]

EXPERIMENTAL RESULTS

All pipettes and volumetric flasks used were calibrated. The burettes used were either furnished with a Bureau of Standards certificate or were equal to their specifications. Two special burettes were used: 10 ml.—divisions 0.05 ml., and 5 ml.—divisions 0.01 ml. Sodium thiosulfate solution was standardized against potassium bromate and potassium bi-iodate. Determinations are recorded in Tables I and II.

The *corrected oxygen values* using values in Tables II (*a*) and I (*a*) are as follows:

March 13, 1933.—0.035 + 0.006 blank = 0.041
0.037 + 0.006 blank = 0.043
March 15, 1933.—0.034 + 0.006 blank = 0.040

The *corrected oxygen values* using values in Tables II (*b*) and I (*b*) are:

March 15, 1933.—0.037 + 0.005 blank = 0.042
0.036 + 0.005 blank = 0.041

Since 1.92 ml. of bi-iodate solution were added in all cases in the results of Tables II (*b*) and I (*b*), the corrected oxygen values could have been obtained as follows using the figures in Tables II (*b*) and I (*b*):

5.28 ml. $Na_2S_2O_3$ − 1.45 ml. $Na_2S_2O_3$ = 3.83 ml. $Na_2S_2O_3$ or 0.042 ml. O_2 per liter
5.19 ml. $Na_2S_2O_3$ − 1.43 ml. $Na_2S_2O_3$ = 3.76 ml. $Na_2S_2O_3$ or 0.041 ml. O_2 per liter

Thus far we have considered the degree of correspondence between the oxygen values obtained by the standard Winkler method and by the modified bi-iodate procedure. Before taking up specifically the

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[1] Kolthoff and Furman, *loc. cit.*, p. 350.

[2] C. H. Fellows and S. J. Meisenburg, "The Determination of Dissolved Oxygen," Paper for Boiler Feedwater Session, Am. Soc. Mechanical Engrs., Annual Meeting, New York City, December, 1933.

effect of oxygen dissolved in the Winkler reagents, we wish to show further the utility of the bi-iodate method:

First, the method enables one to obtain precise oxygen values in spite of considerable variations in the type of starch used and in the temperature at which the titration is performed. There is no special need for considering the variations due to different starches since very sensitive starches can readily be obtained, but temperature control is not always easily obtained in practice.

TABLE I.—BLANK DETERMINATIONS: SENSITIVITY OF IODINE-STARCH-THIOSULFATE REACTION ON DEAERATOR EFFLUENT.

500-ml. sample.
1 ml. arrowroot starch; 0.00096 N $KH(IO_3)_2$; 0.000978 N $Na_2S_2O_35 \cdot H_2O$

Amount of Reagents, ml.	Amount of $KH(IO_3)_2$, ml.	Amount of $Na_2S_2O_3 \cdot 5H_2O$, ml.	Amount of Oxygen per liter, ml.
(*a*) DIRECT ADDITION OF BI-IODATE SOLUTION TO THE APPEARANCE OF BLUE COLOR.			
March 15, 1933:			
2	0.53	....	0.0056
4	0.54	....	0.0058
6	0.58	....	0.0062
b) ADDITION OF EXCESS BI-IODATE SOLUTION AND BACK TITRATION WITH THIOSULFATE TO DISAPPEARANCE OF BLUE COLOR.			
March 15, 1933:			
2	1.92	1.45	0.0047
2	1.92	1.53	0.0049

TABLE II.—OXYGEN DETERMINATION ON DEAERATOR EFFLUENT.

Amount of Reagents, ml.	Volume of Sample, ml.	Amount of $Na_2S_2O_3 \cdot 5H_2O$, ml.	Amount of $KH(IO_3)_2$, ml.	Amount of Oxygen per liter, ml.
(*a*) STRAIGHT TITRATION WITH THIOSULFATE TO DISAPPEARANCE OF BLUE COLOR.				
March 13, 1933: 1 ml. arrowroot starch; 0.0103 N $Na_2S_2O_3 \cdot 5H_2O$				
2	493	0.30	....	0.035
2	500	0.30	....	0.035
2	500	0.30	....	0.035
2	500	0.32	....	0.037
March 15, 1933: 1 ml. arrowroot starch; 0.000978 N $Na_2S_2O_3 \cdot 5H_2O$				
2	500	3.14	....	0.034
(*b*) ADDITION OF EXCESS BI-IODATE SOLUTION AND BACK TITRATION WITH THIOSULFATE TO DISAPPEARANCE OF BLUE COLOR.				
March 15, 1933: 1 ml. arrowroot starch; 0.00096 N $KH(IO_3)_2$; 0.000978 N $Na_2S_2O_3 \cdot 5H_2O$				
2	500	5.28	1.92	0.037
2	500	5.19	1.92	0.036

Second, the method enables one to compensate for unsuspected contamination of reagents and to obtain reasonable approach to accuracy in measurement and even agreement on the dissolved oxygen content of like samples which would ordinarily be at complete variance due to difference in contamination of Winkler reagents.

Third, the effect of the more usual contaminations can be evaluated as a whole, and in most instances differentiated as from reagents and sample, by repeating the determinations on samples of different volumes and with different amounts of reagents. In this manner the dissolved oxygen content of the Winkler reagents can be determined.

Titration Temperature:

The first point may be demonstrated as follows: A sample of deaerator effluent contained, with such oxygen as may have been added through the reagent solutions, about 0.035 ml. of O_2 per liter, titrated at 68 F. by the Winkler method. When the titration was performed at 108 F., the Winkler method gave an apparent zero oxygen value, whereas the bi-iodate method gave the value obtained at the lower temperature:

April 20, 1934

500 ml. samples; 2 ml. Baker's soluble starch: 0.97 ml. of 0.0100 *N* $KH(IO_3)_2$; 0.0107 *N* $Na_2S_2O_3 \cdot 5H_2O$
2 ml. Winkler reagents

(*a*) *Titration at 68 F.:*

Winkler method.—0.31 ml. thiosulfate equals 0.037 ml. of O_2 per liter.

Bi-iodate method.—1.24 ml. thiosulfate on regular sample minus 0.90 ml. thiosulfate on blank equals 0.34 ml. which is equivalent to 0.04 ml. of O_2 per liter.

(*b*) *Titration at 108 F.:*

Winkler method.—No blue color, apparent oxygen concentration zero. 0.20 ml. $KH(IO_3)_2$ required to give a blue color on fixed sample. 0.50 ml. $KH(IO_3)_2$ required to give a blue color on blank. Oxygen concentration equals 0.034 ml. of O_2 per liter, the equivalent of 0.30 ml. of thiosulfate.

Bi-iodate method.—1.13 ml. thiosulfate on regular sample minus 0.82 ml. thiosulfate on blank equals 0.037 ml. of O_2 per liter, the equivalent of 0.31 ml. of thiosulfate.

Reagent Contamination:

The second point concerning the variance in Winkler reagents may be demonstrated as follows: A sample of deaerator effluent contained, with such oxygen as may have been added through the reagent solutions, about 0.03 ml. of O_2 per liter (to be demonstrated). One set of Winkler reagents, used in the previous work in this paper and hereafter called set A, gave a blue color when the conventional test was made. Under the same conditions, another set of reagents to be called set B did not give any blue color. The oxygen value of the particular deaerator effluent was determined by the modified bi-iodate method:

March 20, 1933

500-ml. sample; 2 ml. of Winkler reagents; 1 ml. arrowroot starch solution; 0.000978 *N* $Na_2S_2O_3 \cdot 5H_2O$; 0.00096 *N* $KH(IO_3)_2$

	REAGENTS	AMOUNT OF $Na_2S_2O_3 \cdot 5H_2O$, ML.	AMOUNT OF $KH(IO_3)_2$, ML.	AMOUNT OF OXYGEN PER LITER, ML.
Regular sample....	Set A	3.87	1.92	
Blank sample......	Set A	1.19	1.92	
		3.87 − 1.19 ml. $Na_2S_2O_3$		0.029
Regular sample....	Set A	4.03	1.92	
Blank sample......	Set A	1.28	1.92	
		4.03 − 1.28 ml. $Na_2S_2O_3$		0.030
Regular sample....	Set B	3.60	3.84	
Blank sample......	Set B	0.45	3.84	
		3.60 − 0.45 ml. $Na_2S_2O_3$		0.034
Regular sample....	Set B	3.63	3.84	
Blank sample......	Set B	0.62	3.84	
		3.63 − 0.62 ml. $Na_2S_2O_3$		0.033

Without using the modified bi-iodate method the procedure would have been as follows with set B reagents:

	REAGENTS	AMOUNT OF $Na_2S_2O_3 \cdot 5H_2O$, ML.	AMOUNT OF OXYGEN PER LITER, ML.
Regular sample................	Set B	0.0 (no blue color)	0.0

Proceeding as in Table I (*a*), 3.53 and 3.28 ml. of 0.00096 *N* $KH(IO_3)_2$ were added to blanks to produce a blue color giving the sensitivity as 0.038 and 0.035 ml. of O_2 per liter. Determining oxygen according to conventional procedure upon a regularly treated sample gave no blue color; however, upon the subsequent addition of 0.75 ml. of 0.00096 *N* $KH(IO_3)_2$ a blue color appeared corresponding to an equivalent oxygen addition of 0.008 ml. of O_2 per liter. The corrected oxygen value is therefore $0.037 - 0.008 = 0.029$ ml. per liter. It would appear from the above table that the set B reagents possessed an appreciable iodine absorptive capacity.

Dissolved Oxygen Content of Winkler Reagents:

In Tables I (*a*) and (*b*) we have determined the oxygen concentration indicated as "zero" limited by the sensitivity of test procedure to be of the order of 0.005 ml. of O_2 per liter. On the other hand, considering the third point, the Winkler reagents impart oxygen to the sample. Experiments were undertaken to measure the oxygen content of our Winkler reagents in actual oxygen determinations on deaerator effluent as well as by experiments in which the oxygen concentration in the deaerator effluent was further reduced by the use of a degasifier, an apparatus originally built for the removal of carbon dioxide but equally applicable for the removal of any dissolved gases.

DISCUSSION OF EXPERIMENTAL DATA

Although the precision expected in the Winkler determination of dissolved oxygen should be quite high using calibrated apparatus and good volumetric technique, the final precision attained is largely dependent upon the ability of the individual to judge the end point of the iodine-starch-thiosulfate reaction. A compromise must be effected between decreasing the normality of the thiosulfate solution employed, using ordinary volumetric apparatus, and maintaining the normality approximately at the conventional 0.025 *N* $Na_2S_2O_3$, using micro-

TABLE III.—DETERMINATION OF OXYGEN CONTENT OF REAGENTS.

60 F.; 2 ml. Baker's soluble starch; 0.0100 *N* $KH(IO_3)_2$; 0.0107 *N* $Na_2S_2O_3 \cdot 5H_2O$

Amount of Reagents, ml.	Amount of Oxygen per Liter, ml.			
	Winkler Method		Bi-iodate Method	
	Total in 500-ml. Sample	Total in 250-ml. Sample	Total in 500-ml. Sample	Total in 250-ml. Sample
(*a*) DEAERATOR EFFLUENT WITHOUT PRIOR TREATMENT.				
April 23, 1934:				
1	0.013 0.015	0.019 0.022	0.013 0.015	**0.022** **0.025**
2	0.021 0.020	0.034 0.034	0.022 0.021	**0.036** **0.034**
5	0.040 0.043	0.069 0.067	....	....
ml. of O_2 per liter added per 1 ml. of reagents	0.007	0.012	0.007	**0.012**
ml. of O_2 per liter in original sample	0.007	0.009	0.007	**0.012**
April 24, 1934:				
1	0.017 0.017	0.022 0.025	....	....
2	0.021 0.023	0.034 0.034	....	....
5	0.041 0.044	0.072 0.069	....	....
ml. of O_2 per liter added per 1 ml. of reagents	0.006	0.012	....	....
ml. of O_2 per liter in original sample	0.012	0.011	....	....
(*b*) DEAERATOR EFFLUENT AFTER PASSAGE THROUGH DEGASIFIER—"ZERO" OXYGEN WATER.				
April 25, 1934:				
1	**0.008** **0.010**	**0.009** **0.011**	**0.010** **0.010**	**0.011** **0.011**
2	**0.015** **0.016**	**0.020** **0.022**	**0.016** **0.017**	**0.020** **0.022**
ml. of O_2 per liter added per 1 ml. of reagents	0.008	0.011	0.007	0.010
ml. of O_2 per liter in original sample	0.000	0.000	0.003	0.001

burettes or pipettes. The use of 0.001 *N* $Na_2S_2O_3$ is of course completely inadvisable for routine work. For accurate work, approximately 0.01 *N* solutions with micro-burettes would seem the most appropriate combination. The 0.01 *N* $Na_2S_2O_3$ should be made up freshly each time from 0.1 *N* solution.

It is interesting to compare our values for the oxygen introduced from the Winkler reagents, given in Table III, with the values reported by other observers:

OBSERVER	AMOUNT OF REAGENTS, ML.	AMOUNT OF SAMPLE, ML.	AMOUNT OF OXYGEN, ML. O_2 PER LITER	TEMPERATURE, DEG. FAHR.	STARCH
C. H. Fellows and S. J. Meisenburg[a]	1	250	0.008	50–64	Arrowroot
	2	250	0.019	50–72	Arrowroot
J. R. McDermet[b]	2	500	0.015	50–60	Arrowroot
	2	500	0.013	50–60	Baker's soluble
J. D. Yoder and A. C. Dresher[c]	1	280	0.015	60	Arrowroot
	1	280	0.013	60	Baker's soluble

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[a] C. H. Fellows and S. J. Meisenburg, *loc. cit.*

[b] J. R. McDermet, private communication.

[c] J. D. Yoder and A. C. Dresher, "The Winkler Test for Determining Oxygen in Boiler Feed Water," *Combustion*, Vol. 5, No. 10, pp. 18–22 (1934).

Our experience with the determination of traces of dissolved oxygen has shown that it is not advisable to assume fixed values for the sensitivity of starch as a function of temperature, or for the oxygen content of Winkler reagents. The use of methods that consider the magnitude of these corrections in each actual determination would seem preferable. We recommend the following procedures:

(*A*) Collect two 500-ml. samples in tandem. If the addition of starch does not show a blue color after fixing with 1 and 2 ml. of reagents, respectively, add 0.5 or 1.0 ml. of 0.01 *N* bi-iodate solution. The corrected oxygen concentration of the samples, using 0.01 *N* thiosulfate, is:

$$0.112\ (2 \times \text{ml. of thiosulfate}_{1\,\text{ml.}} - \text{ml. of thiosulfate}_{2\,\text{ml.}}).$$

(*B*) Collect one 250-ml. and one 500-ml. sample in tandem. If the addition of starch does not show a blue color after fixing each with either 1 or 2 ml. of reagents, add 0.5 or 1.0 ml. of 0.01 *N* bi-iodate solution. After fixing, add 250 ml. of untreated sample, collected from the same source and at the same time as the treated samples, to the 250-ml. fixed sample. The corrected oxygen concentration of the samples, using 0.01 *N* thiosulfate, is:

$$0.224\ (\text{ml. of thiosulfate}_{500} - \text{ml. of thiosulphate}_{250})$$

By the use of these methods a "zero" oxygen water may be determined as such within a possible variation of approximately

0.005 ml. of O_2 per liter, notwithstanding the presence of oxygen in the Winkler reagents themselves.

Practical determinations may be made by method *B* without the use of bi-iodate, providing a blue color is formed upon the addition of starch; temperatures above 90 F. are definitely not recommended.

SUMMARY

The conventional Winkler method for determining dissolved oxygen has been compared with a modified bi-iodate method at very small concentrations of oxygen. The use of the bi-iodate method has been shown in (1) eliminating the assumption of a constant sensitivity by automatically canceling the sensitivity correction as an element of the determination; (2) obtaining agreement, and practically true determination, on the oxygen value of a sample notwithstanding the use of Winkler reagents of differing degrees of contamination which by the standard procedure would give results considerably at variance. The oxygen content of our Winkler reagents has been determined by the conventional Winkler method and a modified bi-iodate method. Procedures are suggested for determining the true oxygen content of samples in the neighborhood of zero oxygen.

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DISCUSSION

MR. E. J. THERIAULT[1] (*presented in written form*).—The authors have undertaken the difficult task of determining a constituent present in amounts less than 0.1 p.p.m. by the use of a volumetric procedure. As a rule, the determination of such vanishingly small amounts of any ingredient in water would be based on the more delicate colorimetric procedures. Within this inherent limitation, the technique adopted appears to be the best that can be devised under the circumstances.

Manganous chloride is used instead of the customary manganous sulfate solution. Working with a manganous sulfate solution of standard strength (480 g. of $MnSO \cdot 4H_2O$ per liter) we have recently shown that this solution contains 2.2 p.p.m. of dissolved oxygen per liter when saturated with atmospheric oxygen at 20 C. and a total pressure of 760 mm. The corresponding alkaline-iodide solution appears to be practically devoid of dissolved oxygen (less than 0.1 p.p.m.). These values are much lower than those presented in Table III of the paper. The discrepancy may be accounted for by the presence of traces of manganic salts in the manganous solutions, unless a striking difference exists between the solubility of dissolved oxygen in manganous sulfate and manganous chloride solutions of approximately equivalent strength.

No account appears to have been taken of the possible effect of iron on the Winkler procedure.

It is suggested that a more accurate method for traces of dissolved oxygen might be based on the extraction procedure described by Theriault and McNamee (*Sewage Works Journal*, Vol. 6, pp. 413–422 (1934)) or on the now practically forgotten colorimetric procedure described by Black and Phelps in their report on the "Discharge of Sewage in New York Harbor."

MR. MAX HECHT[2] (*presented in written form*).—The authors deserve commendation for their success in adapting some principles of analytical chemistry to their problem and for the interesting use of the balancing-out method of eliminating errors. A concise description of the manipulative procedure for the modification of the Winkler method would be of interest. The authors refer to a laboratory apparatus designed primarily for the elimination of CO_2 from water which they used in the production of "zero" oxygen water. The details of this apparatus and the procedure used would also be of interest.

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[2] Consultant Chemical Engineer, Pittsburgh, Pa.

It appears to the writer that in a method proposed to give significant values to what formerly were termed traces of O_2, the numerical errors of the method should be reduced to a very small percentage of the uncorrected values. The volume of sample taken should be such that the derived answer is found by division. The titration should be made in water free from interfering substances. Such changes in the procedure will divide automatically the errors resulting from manipulative inconsistencies and the personal equation of the observation of the end point of the titration. It is apparent that in the evolution method used to determine the "zero" oxygen water, the authors met the requirements just stated.

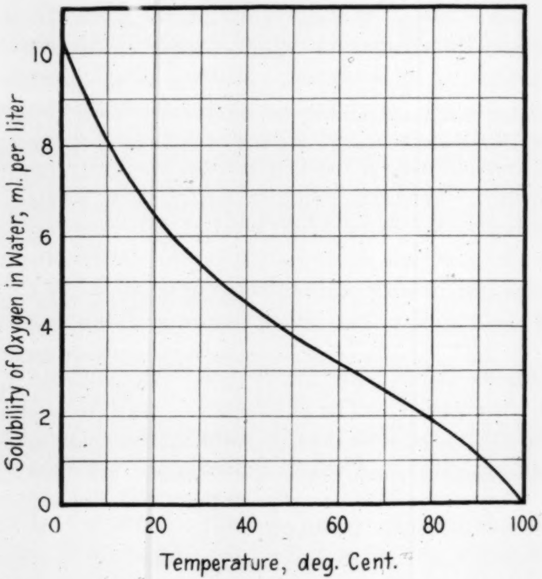

FIG. 1.—Solubility of Oxygen in Water.

Calculated from data of Bohr and Bock, *Annalen der Physik*, Vol. 44, p. 318 (1891).
Total pressure of O_2 plus water vapor = 760 mm. of mercury at 32 F.

It is customary in evaluating the results obtained in an analytical method to observe how they conform to values which may be considered conservatively as fundamental or standard reference values. Mr. D. S. McKinney and the writer have recalculated the values for the solubility of oxygen in water at various temperatures, from the data available which are believed to be the most reliable, and the results are presented in the accompanying Fig. 1. The values of Bohr and Bock were determined by an evolution and gasometric method which should be quite accurate through most of the range of oxygen concentrations indicated, and these values should serve as a satisfactory yardstick to measure the accuracy of any analytical method for dissolved oxygen. The writer believes that the simplest and most satisfactory way to indicate the accuracy of the Schwartz and Gurney method will be to compare the results obtained for a series of samples saturated with oxygen at atmospheric pressure, and at various temperatures, with the values given in Fig. 1. A comparison of the writer's determined values and the Bohr and Bock values are shown in the accompanying Fig. 2.

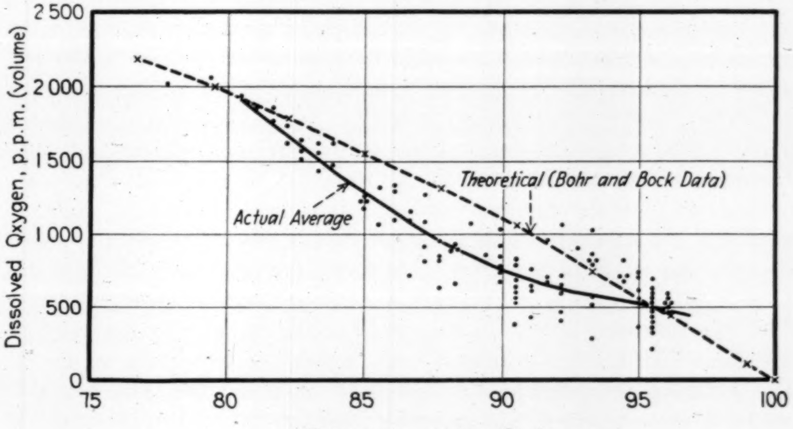

FIG. 2.—Solubility of Oxygen in Water.

Curves referred to partial pressure O_2 plus water vapor = 760 mm. of mercury at 32 F.

It is interesting to note that in a power plant survey concerning corrosion, the values secured for dissolved oxygen[1] by the American Public Health Association Winkler method were not in good agreement with the values of Bohr and Bock shown in Fig. 1. This is most probably due to inaccuracies in the analytical method or to failure to reach equilibrium, and points to the necessity for a more dependable and more accurate procedure for dissolved oxygen, such as Schwartz and Gurney are attempting to obtain.

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[1] C. W. E. Clark, "Boiler-Room Performance and Practice of Colfax Station, Duquesne Light Co.," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 44, p. 242 (1922), see Table 8.

The writer has always been much interested in the determination of dissolved oxygen, especially with regard to its effect on the rate of corrosion; however, it should be noted that in this corrosion problem oxygen is only one of the prime offenders. It is possible that in many cases carbon dioxide or other gases may be equally important.

MR. J. D. YODER[1] (*presented in written form*).—The presentation by Schwartz and Gurney is a valuable contribution towards the establishment of methods and conditions whereby small quantities of dissolved oxygen may be determined with great accuracy. Attention is directed to a number of influences which may cause errors in the determination of oxygen by the Winkler method which are quite appreciable when attempt is made to measure oxygen of boiler feed water in such small quantities as are demanded by modern practice.

They set forth methods whereby oxygen can be determined accurately in the presence of many interfering substances. The accuracy of the methods advanced seems to be established by close agreement of their various tests reported. One may well question, however, if operators in general would be equally successful in cancelling out the effect of the various influences if the methods described were applied.

The inference might be drawn from the paper that the reagents are unavoidably contaminated with impurities which interfere with the accurate determination of dissolved oxygen. Mr. Dresher, with whom I am associated, has carefully investigated this subject and has found no difficulty in securing chemical reagents of satisfactory purity. Contamination of the sodium hydrate - potassium iodide solution has been caused by sulfides extracted from rubber stoppers, but this can be avoided by the use of paraffin-coated glass stoppers. Unquestionably starches have different sensitivities, which vary with the temperature, but fortunately the characteristics of individual starches when once determined are so constant that small quantities of oxygen may be accurately determined by observing the temperature at which the blue color forms without using the sodium thiosulfate titration.

It might be contended that the oxygen in the reagents is a variable quantity and can therefore not be satisfactorily corrected by a constant. This, however, is not necessarily true. It is probable that the oxygen in the reagents can be considered reasonably constant in which event a corrective factor can easily be applied. The probability is that the reagents are always saturated with oxygen either when freshly prepared or after long usage.

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[1] Secretary and General Manager, Cochrane Corp., Philadelphia, Pa.

Mr. Dresher has found[1] that the addition of the manganous chloride and the sodium hydrate plus potassium iodide to distilled water to make up two of the reagents used in the Winkler method reduces the oxygen content from amounts of 4 to 6 ml. per liter in the distilled water to an average of 2.1 ml. per liter in the two solutions. This effect appears to be due to the lower solubility of the oxygen in the highly concentrated solutions. It would be very useful to determine directly, by some method such as the Bunsen method, the concentration of dissolved oxygen in the solutions used in the Winkler method. This information would make it possible to evaluate exactly the amount of oxygen added per milliliter of reagent.

If higher concentrations of reagents should decrease the solubility of oxygen, this would account for the generally lower determinations reported by Schwartz and Gurney than the findings of Mr. Dresher because he used 400 g. of manganous chloride per liter and 420 g. sodium hydrate plus 100 g. of potassium iodide per liter as compared with their solutions 425 g. manganous chloride and 700 g. potassium hydrate plus 150 g. potassium iodide per liter, respectively. Even though different concentrations of reagents were used, it is noticeable that the four independent investigators referred to in the paper agreed within approximately the limits of accuracy defined.

It is noticeable that Schwartz and Gurney's tests on the determination of oxygen in reagents were made in April, 1934, whereas their tests on the use of potassium bi-iodate were made more than a year earlier and that in discussing the application of procedure *B* in the section relating to oxygen in reagents they state "Practical determinations may be made by method *B* without the use of bi-iodate." From this one might infer that the potassium bi-iodate is of minor importance and that it is not depended upon to cancel out the effect of possible contamination of the water supply. In any event it is doubtful if this method would be accurate in cancelling the effect of all contamination as nitrites and possibly other substances.

The practical limit of accuracy in making these determinations is specified as 0.005 ml. which is only 7 parts in a billion. If a double titration is made and these errors are in opposite directions, the accuracy may not exceed 0.01 ml. per liter. If again the oxygen in the reagents is determined by titration of two different size samples, there is the further possibility of error on account of the two samples even though taken consecutively, not actually containing identical quantities of oxygen. If such error is added to the possible error resulting from double titration, it is conceivable that the average operators would make appreciable errors in the application of these methods.

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[1] J. D. Yoder and A. C. Dresher, "The Winkler Test for Determining Oxygen in Boiler Feed Water," *Combustion*, Vol. 5, No. 10, April, 1934, p. 18.

It would seem that the better practice would be to make interference tests to insure the absence of interfering substances, make the tests under standard conditions and apply correction factors that can be determined with exceeding care in the laboratory; only after it is determined that interferences cannot be avoided should corrections be made in the field to offset them. In the latter event the methods proposed by Schwartz and Gurney appear very well adapted.

In my opinion this Society can perform a useful service if it establishes standard conditions including standard strengths of reagents for making determinations of small quantities of oxygen to establish correction factors to be applied for these standard conditions. The average operator I believe will obtain more accurate results in following such a standard routine of practice than if he attempts to make his own corrections for all possible interfering substances, for the latter requires a very high order of skill to make accurate determinations.

MR. J. R. MCDERMET.[1]—I have had the good fortune, not of collaborating in the preparation of this paper, but of knowing in detail of the data during its preparation and of checking independently in a different laboratory the results obtained. The method has proved rather uncannily accurate, barring, of course, the use of grossly contaminated water samples. The procedure is such that the errors due to the preparation of titrating solutions are minimized. It is sometimes quite difficult to get commercial chemicals regardless of the stated purity or price which yield satisfactory and consistent values. It is also true that the average operator in industry is able to get contamination by careless handling of apparatus and mixing of solutions. In our own laboratory, we have consistently obtained values which indicate an accuracy of 7 per cent for values below 0.10 ml. per liter, provided, of course, this 7 per cent is large enough to have any numerical significance. The laboratory is a commercial laboratory of a manufacturer and not a research laboratory, but has had extensive experience in this class of work.

These values mentioned were obtained by either synthetic solutions or by the method of mixtures with laboratory deaerated water and aerated water which had been carefully standardized for dissolved oxygen by another method, notably an evolution method. Naturally the percentage value given fails when the percentage amounts to less than 0.003 ml. per liter. The limitation is then in the indicator which is no longer sufficiently sensitive. My viewpoint, therefore, is that in practical work where contamination of the initial sample is not gross, the method proposed by Schwartz and Gurney, while not chemically new in theory, is essentially very useful.

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[1] Chief Engineer, Elliott Co., Jeanette, Pa.

It is apparently true that a boiler water cannot be judged from the standpoint of safety on its dissolved oxygen content alone; other factors, such as the pH value are of equal or more importance. The average power plant operator, however, insists on the maximum in mechanical deaeration whether he needs this or not. Since, at the present time, it is not possible to determine just how much deaeration is needed the operator naturally plays safe; therefore, a test method accurate at low concentrations of oxygen is valuable for commercial judgment.

MR. T. H. DAUGHERTY.[1]—Of the two procedures described by Schwartz and Gurney, procedure *B* appears to be the most practical and is the one actually in use at the Louisiana Steam Generating Corporation. Exact instructions for the use of the test are not given by the authors, and the following summary presents these instructions as used by the Hall Laboratories, Inc.:

> One 500-ml. and two 250-ml. samples are collected as specified in the paper. The 500-ml. and one of the 250-ml. samples are each fixed in the normal Winkler manner, adding the same quantities of reagents to each, and adding 1 ml. of potassium bi-iodate solution to each after fixing.
>
> The 250-ml. fixed sample and the 250-ml. unfixed sample are both poured into an empty 500-ml. flask. Exactly 500 ml. of the sample so prepared are measured into an 800-ml. beaker and titrated in the usual manner with freshly standardized 0.01 *N* sodium thiosulfate. Exactly 500 ml. of the 500-ml. fixed sample are similarly measured and titrated.
>
> This second titration value minus the first titration value is equal to the number of milliliters of 0.01 *N* sodium thiosulfate equivalent to the oxygen initially present in 250 ml. of the sample, free from the errors normally present in the Winkler titration.
>
> The oxygen content can then be calculated, since one ml. of 0.01 *N* sodium thiosulfate, on the basis of a 250 ml. sample, is equivalent to 0.32 p.p.m. of oxygen or to 0.224 ml. oxygen per liter (standard conditions).
>
> The necessity for accurate pipettes and finely calibrated burettes is apparent, since 0.05 ml. of 0.01 *N* sodium thiosulfate is equivalent to 0.016 p.p.m. oxygen or 0.011 ml. oxygen per liter and 0.01 ml. to 0.003 p.p.m. oxygen or 0.002 ml. oxygen per liter, while the accuracy of the method is given as 0.005 ml. oxygen per liter or 0.007 p.p.m. oxygen.

MESSRS. M. C. SCHWARTZ[2] AND W. B. GURNEY[3] (*authors' closure by letter*).—The authors believe that the points raised in the preceding discussions may best be met by an outline of procedure as actually adopted in power station practice:

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[1] Hall Laboratories, Inc., Pittsburgh, Pa.

[2] Research Chemist, Louisiana Steam Generating Corp., Baton Rouge, La.; and Boiler Feedwater Fellow, Institute of Industrial Research, Louisiana State University, Baton Rouge, La.

[3] Efficiency Engineer, Louisiana Steam Generating Corp., Baton Rouge, La.

Method A.—Four samples of which two (500-ml. Erlemeyer flasks in tandem containing three-holed rubber stoppers for an inlet and outlet tube as well as a solid glass plug for reagent addition) are collected in the usual manner and two of which may be collected in the open. Then add the following reagents in the order and quantities listed below shaking well after each addition of reagent:

SAMPLE T_1	SAMPLE T_2	SAMPLE B_1	SAMPLE B_2
1 ml. $MnCl_2$	2 ml. $MnCl_2$	1 ml. alk. KI	2 ml. alk. KI
1 ml. alk. KI	2 ml. alk. KI	1 ml. H_2SO_4	2 ml. H_2SO_4
1 ml. H_2SO_4	2 ml. H_2SO_4	1 ml. $MnCl_2$	2 ml. $MnCl_2$
1 ml. $KH(IO_3)_2$	1 ml. $KH(IO_3)_2$	1 ml. $KH(IO_3)_2$	1 ml. $KH(IO_3)_2$

Take 500 ml. for titration with 0.01 N thiosulfate.

Then $2\,(T_1 - B_1) - (T_2 - B_2)$ = milliliters of oxygen in sample or for 500-ml. samples.

$0.112 \times [2(T_1 - B_1) - (T_2 - B_2)]$ = milliliters of oxygen per liter in sample.

Method B.—Two samples (500 ml. and 250 ml. in tandem containing three-holed rubber stoppers for an inlet and outlet tube as well as a solid glass plug for reagent addition) are collected in the usual manner. Then add the following reagents in the order and quantities listed below:

SAMPLE T_{500}	SAMPLE T_{250}
2 ml. $MnCl_2$	Same as for T_{500}
2 ml. alk. KI	Same as for T_{500}
2 ml. H_2SO_4	Same as for T_{500}
1 ml. $KH(IO_3)_2$	Same as for T_{500}

Add 250 ml. of untreated water to T_{250} and titrate both 500-ml. samples with 0.01 N thiosulfate. Then $0.224\,(T_{500} - T_{250})$ = milliliters of oxygen per liter in sample.

From the above formulae the following points may readily be appreciated.

1. Operators now using the straight Winkler test for oxygen may use Method B, in many cases, and still obtain accurate results without the use of the bi-iodate solution.

2. If, however, a blue color is not obtained in either the 500-ml. or 250-ml. sample in Method B, it will become necessary to add the bi-iodate solution to detect the dissolved oxygen present.

The use of the bi-iodate solution is not at all involved, and it is the simplest "oxygen solution" to prepare. Any operator who can carry out the straight Winkler procedure should find no difficulty in using bi-iodate.

Method B (using bi-iodate) has now been used at the Louisiana Steam Generating Corp. for nearly a year with satisfactory results for determining deaerator performance.

A COMPARISON OF THE METHODS FOR TESTING INSULATING MATERIALS FOR POWER FACTOR AND DIELECTRIC CONSTANT AT 1000 CYCLES

By W. B. Kouwenhoven[1] and L. W. Marks[2]

Synopsis

This paper discusses the series resistance and the parallel resistance bridges which have been proposed as A.S.T.M. methods for the measurement of dielectric characteristics at 1000 cycles. The possibility of using the balanced Schering bridge for the same purpose was also investigated. These methods were studied both from the theoretical and practical viewpoints. Measurements were made upon a number of samples by all three methods and the results compared. The authors recommend the use of the substitution method and a Wagner grounded bridge. They point out that the balanced Schering bridge offers a single piece of apparatus that may be satisfactorily used over a range of frequency from 25 to 1000 cycles.

In the Tentative Method D 150 - 32 T (1)[3] developed by Committee D-9 on Electrical Insulating Materials of the American Society for Testing Materials three methods are set forth for the measurement of the dielectric constant and the power factor of insulating materials at 1000 cycles. The three methods are:

1. The inductance bridge of Rosa and Grover (2).
2. The series resistance bridge of Max Wien (3).
3. The parallel resistance bridge of Rosa (4).

This paper deals with a study of these three methods and also discusses the possibilities of using the balanced Schering bridge as an alternate method of measuring the constants of insulating materials.

In this study the inductance bridge was eliminated at an early stage because the tentative method calls for specimens whose capacitance varies from 1000 to 3500 $\mu\mu$f. for use in this bridge, while in the other two bridge methods specimens of only one-tenth of this size are required. Therefore, the inductance bridge was eliminated from the comparison.

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[1] Professor of Electrical Engineering, Johns Hopkins University, Baltimore, Md.

[2] Student Engineer, General Electric Co., Schenectady, N. Y.

[3] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 838.

The investigation covered a wide field which may be divided into the following parts:

1. The series resistance, the parallel resistance and the Schering bridges were studied from a theoretical viewpoint to determine their inherent errors. This study resulted in the decision to recommend the parallel substitution method as the most satisfactory plan of operation for all three bridges.
2. Their shielding was investigated.
3. The sensitivities of the bridges were compared.
4. Where possible each bridge was checked for accuracy against a low power factor standard.
5. The bridges were compared with and without the use of the Wagner ground.
6. A number of specimens covering a wide range of insulating materials were tested both with and without guard electrodes.
7. The effect of the spacing of guard electrodes was measured as a function of specimen thickness.

Before commencing the theoretical study of the bridges from the standpoint of residual errors, effects of stray capacitances, etc., it was necessary to consider the type and test conditions of specimens which were to be tested. The specimens consisted of flat disks of insulating material to which lead foil electrodes could be applied to opposite surfaces. It is well known that the constants of a given sample of insulation vary with the temperature and that if check results are to be secured, the temperature must be held constant. In order to meet this requirement the specimens were placed in a box in which the temperature and humidity were maintained constant. In such a constant temperature container the electrode surfaces on the specimen are within fairly close proximity to the walls of the box and the resulting stray capacitance will introduce an error in the results unless it is taken into account or eliminated. This stray capacitance and its elimination were considered in the analysis of the bridges.

SERIES RESISTANCE BRIDGE

The series resistance bridge shown in Fig. 1 is one of the proposed standard methods. In the commercial bridge of this type the ratio arms R_1 and R_2 are each 5000 ohms, C_s is a standard air capacitor with a range of 50 to 1500 $\mu\mu$f., C_1 is an air capacitor of at least 1000 $\mu\mu$f. The specimen is represented by the capacitance C_x. The resistance R_3 is variable in 1.0-ohm steps. The resistances R_1, R_2 and R_3 and the input and detector transformers are enclosed in a metal box. When the bridge is operated as a grounded bridge, the ground connection (Fig. 1) is that recommended by Field(5).

Using the substitution method the bridge relations are:

$$C_x = C'_s - C_s \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$\text{Power factor} = 2\,\pi f\,(R_3 - R'_3)\,\frac{(C'_s)^2}{(C_x)}\,10^{-12} \quad \ldots\ldots\ldots\ldots (2)$$

where C_x is the capacitance of the specimen in micro-microfarads and C_s and R_3 denote respectively the values of C_s and R_3 when the specimen is in circuit, and their primes the values when the specimen is disconnected.

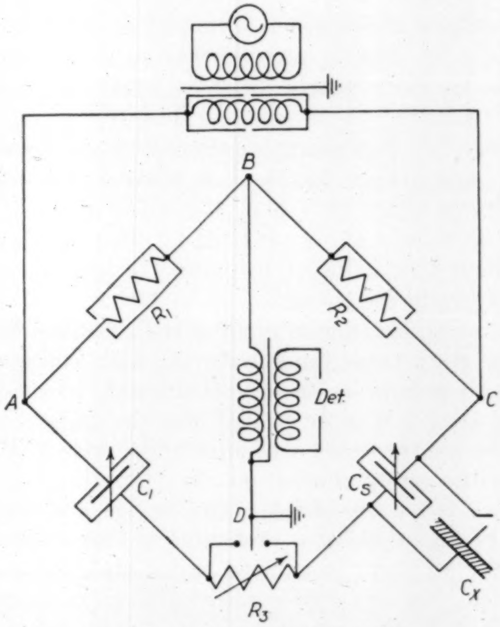

FIG. 1.—Series Resistance Bridge.

The grounding of the bridge at the point D is the best arrangement from the standpoint of the elimination of errors unless double shielding is employed. Siskind(6) has shown that with any other ground connection or a Wagner ground, the stray capacitance from the capacitor C_1, and resistance R_3, and their connecting lead to ground, introduce an error in power factor which is directly proportional to the ratio of this stray capacitance to C_1.

Another source of error in this bridge is the capacitance to ground of the secondary of the input transformer and of the leads connecting the capacitors to the corners of the bridge. In a one to one bridge the effect of this stray capacitance may be eliminated by splitting the secondary shield, Fig. 1, and arranging the leads so as to divide this capacitance equally between the arms *AD* and *CD*. In case this stray capacitance is not equally divided the power factor equation becomes:

$$\text{Power factor} = 2\,\pi f\,(R_3 - R'_3)\,\frac{(C'_s \pm C_3)^2}{C_x}\,10^{-12} \qquad (3)$$

where C_3 represents the unbalanced stray capacitance.

The accuracy of the division of this stray capacitance to ground may be checked by making power factor measurements of a given sample at two or more widely different values of C'_s. If the capacitances are balanced, the results as calculated by Eq. 2 will check at all values of C'_s. For example, a known specimen of capacitance $C_x = 289$ μμf. and power factor $\delta_x = 0.00193$ was measured in a commercial bridge which gave a $\delta_x = 0.00186$ at a $C'_s = 360.5$ μμf. and a $\delta_x = 0.00192$ at a $C'_s = 1192.7$ μμf. This indicated that there was an unbalanced capacitance to ground of approximately 5.5 μμf. across the *CD* arm in the circuit.

An error, which is common to all of the grounded bridges, is that caused by the stray capacitance from the high voltage electrode of the specimen to ground as already mentioned. It may be readily eliminated by using a Wagner ground with the bridge.

In order to use the series resistance bridge with a Wagner ground and eliminate the errors pointed out by Siskind(6), the arrangement shown in Fig. 2 is recommended. This bridge is a conjugate of the arrangement of Fig. 1 and it is preferable to that connection because the load on the oscillator is reduced and a higher voltage is applied to the specimen. In this double shielded series resistance bridge the ratio arms R_1 and R_2, the capacitors C_1 and C_s, and the Wagner ground components C_6, R_6 and R_5, as well as the input and detector transformers, were all enclosed in a grounded metal box. A small fixed mica capacitor C_2 is connected across the ratio arm R_2 so as to keep the resistance R_3 always in the *AD* bridge arm. The value of this capacitor depends upon the quality of capacitor C_1. The capacitor C_1 and the resistance R_3 are each enclosed in separate grounded boxes and the capacitance to ground of their common lead is kept a minimum. The resistance R_3 is at a high potential and its dial handles were brought out through the ground shielding to avoid the effects of body capacitance.

The insertion of a ground shield between the resistance R_3 and capacitor C_1 as used by Sommerman(7) eliminates the stray capac-

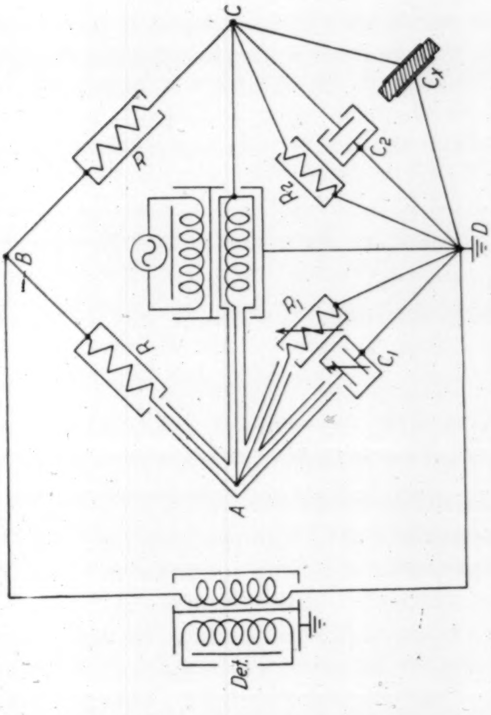
C
R
R
B
A
D
R_1
C_1
R_2
C_2
C_x
Det.

FIG. 3.—Parallel Resistance Bridge.

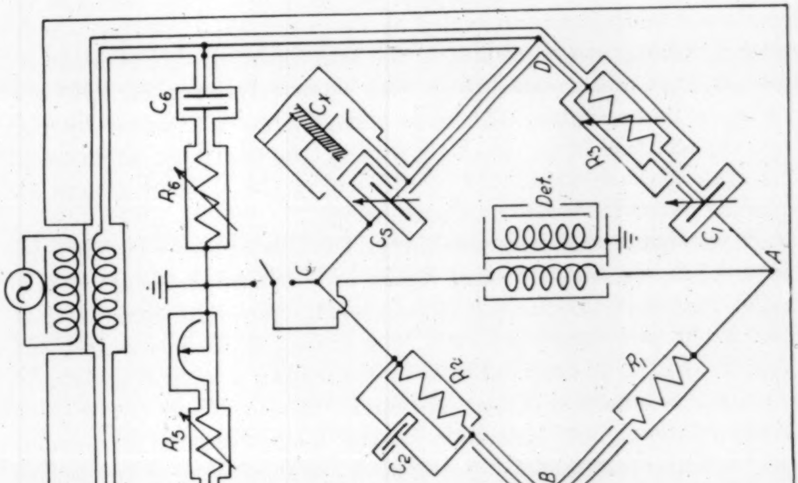
C_6
R_6
C_x
D
R_3
Det.
C_5
C
R_5
R_2
C_2
B
R_1
A
C_1

FIG. 2.—Double Shielded Series Resistance Bridge.

itance between the A and D corners of the bridge and their effect upon the results.

The use of the Wagner ground makes it possible to completely shield the high voltage lead to the specimen and eliminate the capacitance of the leads from the results. It also eliminates the stray capacitance from the high voltage electrode to the specimen container and permits the use of guard electrodes on the specimen if desired.

PARALLEL RESISTANCE ARM BRIDGE

The parallel resistance arm bridge with the A.S.T.M. recommended shielding and ground is shown in Fig. 3. In this bridge the equations are:

$$C_x = C_s - C'_s \qquad (4)$$

$$\text{Power factor} = \frac{10^{12}\,(R' - R)}{2\,\pi f\,C_x R' R} \qquad (5)$$

where C_x = capacitance of the specimen in micro-microfarad,
C'_s = capacitance of C_1 with specimen out,
C_s = capacitance of C_1 with specimen in,
R' = resistance of R_1 with specimen out,
R = resistance of R_1 with specimen in, and
f = frequency in cycles per second.

With the connections as shown in Fig. 3 and using the type of substitution method recommended by the A.S.T.M. Tentative Standard, any residual errors present in the ratio arms are not eliminated. However, in a one to one bridge with accurately balanced residuals their effect is negligible. There is also an error introduced by the stray capacitance from the high side of the specimen to ground. This error may be eliminated by the use of the Wagner ground as explained under the series resistance bridge.

In its commercial form this bridge has the following circuit constants. The resistances R_1 and R_2 are each 10,000 ohms and R_1 is a six-dial resistance box having steps varying from 0.01 ohm to 1000 ohms. The ratio arms are each 1000 ohms and C_2 has a value of 200 $\mu\mu$f. In the commercial form of the bridge, a Wagner ground is permanently connected across the source. This arrangement of placing the relatively low resistances of the Wagner ground in parallel with the capacitors noticeably decreases the sensitivity of the bridge when it is used as a grounded bridge. It is desirable to provide means of readily disconnecting the Wagner ground not only in this bridge but also in all commercial bridges because occasions may arise where it is necessary to operate the bridge grounded.

In using the commercial bridge with specimens of low power factor, the difference between the two values of R_1 with the specimen in and with the specimen out is small. This difference may be increased by the use of 100,000-ohm resistances in R_1 and R_2 and their use increases the difference $R' - R$ one hundred times for a given specimen. Large resistance units in R_1 and R_2 materially increase the sensitivity of the bridge in the range of frequencies used.

BALANCED SCHERING BRIDGE

It has long been recognized that the Schering bridge offers an excellent means for measuring the constants of insulating materials at 60 cycles. Therefore, it was decided to try the bridge at 1000 cycles and to determine its possibilities. The diagram of the bridge used and its shielding are shown in Fig. 4 complete with the Wagner ground. The variable arms are the C_1, R_1 arm and the C_4 arm. The equations using the parallel substitution method are:

$$C_x = C'_s - C_s \qquad (6)$$

$$\text{Power factor} = 2\,\pi f\, R_1\,(C_1 - C'_1)\,\frac{C'_s}{C_x}\,10^{-12} \qquad (7)$$

where C'_s = capacitance of C_4 with specimen out,
C_s = capacitance of C_4 with specimen in,
C'_1 = capacitance of C_1 with specimen out, and
C_1 = capacitance of C_1 with specimen in.

This bridge was operated successfully at both 60 and 1000 cycles. The resistances R_1 and R_2 were each 5000 ohms. The capacitor C_3 was a mica capacitor of 250 $\mu\mu$f. The capacitors C_1 and C_4 were variable, each with a range of 50 to 1500 $\mu\mu$f. For maximum sensitivity C_3 should have a capacitance about 1.5 times the value of C_x. In the Wagner ground the resistance R_5 had a maximum value of 1100 ohms and R_6 11,110 ohms. A small slide wire resistance of a few ohms range was provided for fine adjustment in the R_5 circuit. When the bridge was used at 1000 cycles the Wagner ground mica capacitor C_6 had a value of 0.005 μf. For 60-cycle work an additional mica capacitor of 0.03 μf. was connected in the circuit. The bridge was shielded and was completely enclosed in a grounded metal box.

The balanced Schering bridge of Fig. 4 proved very simple to operate, including the balancing of the Wagner ground. The conjugate arrangement suggested by Siskind(6) was tried. It gave accurate results, but required considerably more time and skill to balance than the bridge of Fig. 4. At 1000 cycles the bridge was supplied with power from the oscillator and the amplifier with telephones was used in the detector circuit, described later. At 60 cycles the supply was furnished by a sine wave generator and the detector was either a vibration galvanometer or a Leeds and Northrup alternating current needle galvanometer separately excited by means of a phase shifting transformer using the method developed by Kouwenhoven and Banos(8). This latter method permits separate balancing of the power factor and capacitance, but where it is used in conjunction with an amplifier in the detector circuit, the operation of the amplifier should be maintained constant.

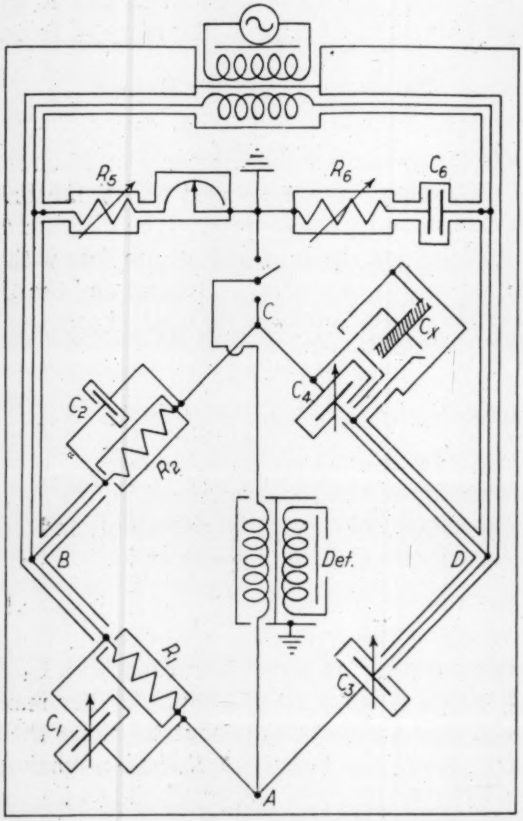

FIG. 4.—Balanced Schering Bridge.

The Schering bridge may be also used as a solidly grounded bridge, but unless guard electrodes with guard balancing equipment(8) are used on the specimen, it possesses many of the disadvantages of the other solidly grounded bridges.

Using the parallel substitution method in the balanced Schering bridge eliminates all residual errors and with the Wagner ground the effects of capacitance from the specimen electrodes to ground are not present. This bridge gave satisfactory results at both 60 and 1000 cycles. It may also be operated at somewhat higher frequencies if desired. It had ample sensitivity at both frequencies.

OSCILLATOR AND DETECTOR

The A.S.T.M. Tentative Method D 150 – 32 T gives one the choice of several types of 1000-cycle sources of supply. For this work the vacuum tube oscillator was chosen, using the Hartley(9) circuit and a No. 210 tube as the oscillator. The output of the oscillator was fed into a simple class A amplifier employing a No. 250 tube. The output of the plate circuit of this tube could be connected to either a tuned air core variable coupling transformer or to a special iron core transformer. In both transformers primary and secondary were separated by shields. With these arrangements a supply of energy at 1000 cycles was available over a voltage range of 5 to 300 volts. The frequency of the oscillator was adjusted to 1000 cycles using a frequency sensitive Wien bridge and was checked by an oscillograph. It was found to be constant to within two cycles over the entire range of load and to be independent of normal changes in plate and filament voltages. The detector circuit consisted of a Western Electric type 104AE shielded output transformer feeding into a two- or three-stage resistance capacity coupled amplifier. The output of the last stage of this amplifier was fed through a one to one transformer and the telephones were attached to the secondary.

SENSITIVITY COMPARISON

There are several methods available for determining the sensitivity of bridges. In this work the series resistance bridge and the parallel resistance bridge were available in commercial form, and the balanced Schering bridge was built up as shown in Fig. 4 from laboratory equipment.

In measuring the relative sensitivity of these bridges, they were balanced using a value of capacitor C'_s of 300 $\mu\mu$f. Then assuming that the average size of specimen has a capacitance of 200 $\mu\mu$f., the change in the power factor balancing arm corresponding to a change in the power factor of the specimen of 0.0008 was calculated. The power factor balancing unit was then upset by this amount and the resulting noise in the telephones reduced to extinction by an audibility meter. Then the power factor balance was restored and the capacitance balance upset by 0.6 $\mu\mu$f. in C'_s and the test repeated. In the case of the two commercial bridges, 30 volts at 1000 cycles was applied to their input terminals, and in the Schering bridge the same voltage was applied directly to the B and D bridge terminals. In all tests the amplification of the detector output was held constant. The results of a set of typical sensitivity measurements using all three bridges solidly grounded and with Wagner grounds disconnected are given below. A high reading of the audibility meter indicates high sensitivity:

	SETTING OF AUDIBILITY METER	
BRIDGE	FOR POWER FACTOR UNBALANCE	FOR CAPACITANCE UNBALANCE
Commercial series resistance bridge	400	1300
Commercial parallel resistance bridge	29	74
Laboratory Schering bridge	200	650

In the commercial parallel-resistance bridge the Wagner ground is solidly connected across the source so that when used as a grounded bridge it becomes a shunting impedance on the R_1 and R_2 arms. Using the bridge as it is designed commercially, and grounded, its sensitivity fell so that for the given power factor unbalance the audibility meter read 11 and for the capacitance unbalance 29. Using it with Wagner ground disconnected and 100,000-ohm boxes in R_1 and R_2 instead of the 10,000-ohm units furnished in the commercial bridge, its sensitivity reading was 150 for power factor upset and 500 for capacitance upset. Experience in operation and other sensitivity tests confirm the above results.

ACCURACY STUDY OF THE BRIDGES

In the Engineering Laboratories at The Johns Hopkins University there is available the Low Power Factor Standard of Kouwenhoven and Berberich(10). Briefly this consists of a shielded and guarded loss-free capacitor connected in series with ideally shielded resistors. In this investigation two resistors were used, giving at 1000 cycles power factors of 0.03322 and 0.00325, respectively. The capacitance of the low power factor standard is 517.5 $\mu\mu$f., which is larger than that of the specimens, but well within the range of the three bridges.

Only bridges employing the Wagner ground may be readily checked against the low power factor standard. A special guard circuit with complicated shielding is required for checking a grounded bridge. The double shielded series resistance bridge of Fig. 2, the commercial parallel resistance bridge of Fig. 3 and the balanced Schering bridge of Fig. 4, each equipped with its Wagner ground, were checked against the power factor standard. In these tests the same source of power and detector arrangement were used. The results are given in Table I.

The results show good accuracy to within the sensitivity of the individual bridges. The series resistance and Schering bridges with a sensitivity of the order of 1×10^{-5} in power factor checked the standard in every test to within better than 3×10^{-5} in power factor. In the commercial parallel resistance bridge a variation of 0.01 ohm in R_1 corresponds to a change in power factor of 3×10^{-5} at this value of C_x. This small change in R_1 is difficult to detect at 1000 cycles. The differences between the true and measured values given in Table I are directly attributable to the lower sensitivity of this bridge. Using 100,000-ohm resistance boxes in the R_1 and R_2 arms of this bridge, an excellent check was obtained with the power factor standard.

TABLE I.—RELATION OF MEASURED POWER FACTOR TO STANDARD.

BRIDGE	STANDARD POWER FACTOR	MEASURED POWER FACTOR	MEASURED CAPACITANCE
Series resistance (Fig. 2)	0.03322	0.03322	517.6
	0.00325	0.00322	517.2
Parallel resistance (Fig. 3), 10,000 ohm	0.03322	0.03315	516.9
	0.00325	0.00314	517.2
	0.00325	0.00332	517.8
Parallel resistance (Fig. 3), 100,000 ohm	0.03322	0.03320	516.1
	0.00325	0.00322	516.2
Schering Fig. 4	0.03322	0.03320	517.5
	0.00325	0.003245	517.7

TABLE II.—ELECTRICAL PROPERTIES OF TYPICAL SPECIMENS.

Specimen	Material	Form	Thickness, in.	Dielectric Constant	Power Factor	Hours to Reach Final Value
L-1	Laminated bakelite	Round	0.192	5.93	0.0368	6
L-2	Laminated bakelite	Square	0.192	5.94	0.0367	8
C-1	Clear yellow bakelite	Square	0.1768	7.01	0.0566	8
C-25	Clear blue bakelite	Square	0.1913	3.93	0.00696	..
X-150-2	Fine fabric	Square	0.0638	4.73	0.0200	over 48
Z	Paper laminated	Square	0.1279	5.38	0.0248	6
ZM-264	Black bakelite	Square	0.1188	6.55	0.1175	8

SPECIMENS

Specimens were employed in the further comparison of the bridges. They were used in the study of the solidly grounded and Wagner grounded bridges, in the comparison of the results on guarded and unguarded specimens, and on the effects of guard spacings. The electrodes were of lead foil and on specimens without guards were

4.5 in. in diameter. On guarded specimens the high-voltage electrode was a 6-in. round or square disk, depending upon the sample, and the low voltage electrodes 4.5 in. in diameter. Table II gives a list of typical specimens referred to in this paper and their electrical properties as measured with guard electrodes at 30 C. and 60 per cent relative humidity.

The importance of maintaining constant temperature and humidity of a specimen during a test has already been referred to. A box 7.5 in. square by 5.5 in. high was used to hold the specimens. This box was placed in an oil bath and provided with thermostatic control which maintained the temperature constant to within ±0.1 C. The humidity within the box was held at a constant value by the use of a concentrated ammonium nitrate solution. The constant temperature-humidity chamber is shown in Fig. 5.

In Fig. 5 may also be seen the spring and weight contacts found convenient and satisfactory for the rapid interchange of specimens. The brass weight, about 1 lb., was sufficient to depress the spring and at the same time insure good contact with the guard shield. The latter was removed when measuring specimens with equal electrodes.

Tests were made on the same specimens, using No. 30 B. & S. leads soldered to both upper and lower electrodes and then replacing the leads with the weight and spring contact of Fig. 5. The results are given below on specimen L-1 as measured in two of the bridges after being held at constant temperature and humidity for over 24 hours:

Bridge	Bridge Ground	Contacts	C_x, $\mu\mu$f.	Dielectric Constant	Power Factor
Commercial series resistance	Solid	Fine wire	115.0	6.17	0.0350
Commercial series resistance	Solid	Weight and spring	115.9	6.22	0.0348
Schering	Wagner	Fine wire	110.5	5.93	0.0368
Schering	Wagner	Weight and spring	110.5	5.93	0.0368

Both electrodes on the specimen were 4.5 in. in diameter. The tests on the solidly grounded commercial series resistance bridge, where the measured capacitance includes the capacitance of the high voltage electrode to ground, show that the weight increased this capacitance by nearly 1 $\mu\mu$f. When a Wagner grounded bridge was used, the results show clearly that the capacitance of the specimen and its power factor are exactly the same for both types of connection.

The importance of maintaining specimens at constant temperature cannot be overemphasized. For example, specimen C_1 at 20 C. had a power factor of 0.035 and a dielectric constant of 6.64; at 30 C. these values had increased to 0.056 and 7.01, respectively. This also

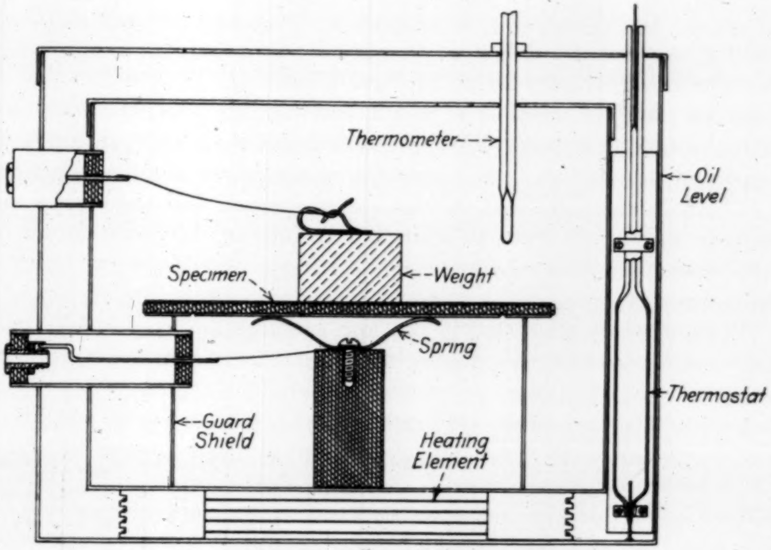
Thermometer
Oil Level
Specimen
Weight
Spring
Thermostat
Guard Shield
Heating Element

FIG. 5.—Specimen Container.

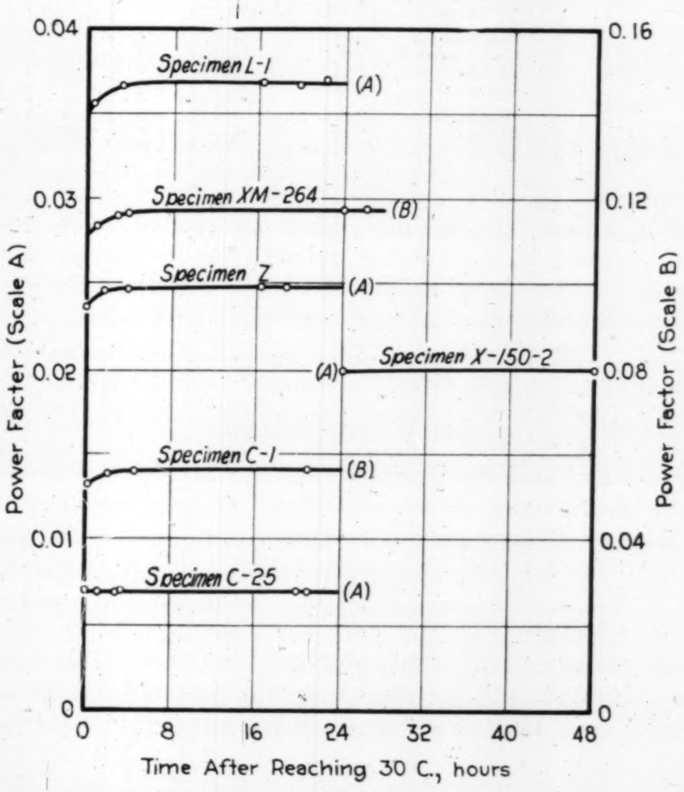

FIG. 6.—Time to Reach Steady Conditions. Room temperature 21 to 23 C.

brings out the desirability of measuring specimens throughout their working temperature range.

The A.S.T.M. Tentative Method D 150 - 32 T provides that a specimen shall be held at constant temperature and humidity for 48 hours before testing. With this requirement in view, a series of measurements was made on a number of specimens at intervals after the thermometer indicated constant temperature, and the results are given in Fig. 6. It may be seen that the change in power factor of the specimens measured was negligible after 6 or 8 hours at constant temperature.

It is recommended that in practice, readings be taken at intervals after a specimen has been placed in a constant temperature container, and that a power factor - time curve be plotted. This will be helpful in determining when the specimen has reached equilibrium.

TABLE III.—DATA FOR SPECIMENS TESTED IN THE CONSTANT TEMPERATURE BOX

Specimen	Bridge	Type of Grounding	$C_x - C_e$	Dielectric Constant, K	Power Factor	Percentage Difference in K
L-1	Series resistance	Solid	115.0	6.17	0.0350	+4.6
	Parallel resistance	Solid	115.0	6.17	0.0360	+4.6
	Schering	Solid	114.7	6.15	0.03588	+4.2
	Parallel resistance	Wagner	109.5	5.885	0.03675	0
	Schering	Wagner	109.8	5.900	0.03690	0
C-1	Series resistance	Solid	146.2	7.23	0.0525	+3.4
	Parallel resistance	Solid	144.5	7.15	0.0521	+2.3
	Schering	Solid	145.2	7.18	0.0507	+2.7
	Schering	Wagner	141.2	6.99	0.0555	0
C-25	Parallel resistance	Solid	76.7	4.07	0.0071	+3.5
	Schering	Solid	76.5	4.06	0.00682	+3.3
	Schering	Wagner	74.2	3.93	0.00696	0

It is also interesting to note that over the range of 25 to 300 volts at 1000 cycles, no differences were found in the constants of the materials tested. Hartshorn(11) obtained similar results on sheets of varnished cloth.

THE WAGNER GROUND

The capacitance from the high voltage electrode to ground causes an error in the solidly grounded bridge. It may be eliminated by the use of the Wagner ground as already mentioned. In Table III are given data for three specimens, L-1, C-1 and C-25 which were tested in the constant temperature box, using fine wires soldered to their 4.5-in. electrodes. In the case of specimen C-25 using the Wagner ground, guard electrodes were employed. The measured value of the specimen capacitance C_x was corrected, using the edge correction C_e of Hoch(12) and the dielectric constant of the specimen calculated from the corrected value. A study of Table III shows clearly that the dielectric constant is too high for specimens measured in any one of the three bridges when solidly grounded. The calculated power factor is also in error in such cases. When, however, the Wagner ground was used, this stray capacitance was eliminated and the results are much more accurate.

GUARD ELECTRODES

A number of experiments were made using guard electrodes to check the edge correction and the effects of surface leakage at 60 per cent relative humidity. In Table IV are given typical results of measurements on four specimens of varying thickness. All of these were measured after having been held at constant temperature for at least 20 hours. Measurements were made using equal 4.5-in. diameter electrodes on both surfaces, and using the same sample with a 6-in. electrode on one side and a 4.5-in. electrode with guard ring opposite. The spacing between the guard ring and electrode was small. In Table IV C_x is the measured value of specimen capacitance. In calculating the dielectric constant with guard ring, the effective radius of the 4.5-in. diameter electrode must be increased by one-half of the spacing between the guard ring and the electrode. With equal electrodes the edge correction C_e was used.

TABLE IV.—TESTS MADE WITH AND WITHOUT GUARDS ON SCHERING BRIDGE WITH WAGNER GROUND.

Specimen	Electrodes	C_x, $\mu\mu$f.	C_e, $\mu\mu$f.	Dielectric Constant	Power Factor	Thickness, in.
C-25	Guards	74.2	....	3.93	0.00696	0.1913
	Equal	76.7	3.6	3.91	0.00705	
X-150-2	Guards	267.2	....	4.74	0.01975	0.0638
	Equal	268.7	4.1	4.73	0.01987	
L-1	Guards	112.9	....	5.95	0.03685	0.192
	Equal	115.3	4.8	5.93	0.0368	
C-1	Guards	142.9	....	7.01	0.0566	0.1768
	Equal	146.4	5.2	6.99	0.0555	

The results of Table IV show that over the rather wide range of thickness and dielectric constant, Hoch's correction gives good results, and that surface leakage was negligible.

In applying guards to a specimen, care must be taken to keep the spacing between the guard and the electrode small, otherwise errors will be introduced by the non-uniform flux distribution in this space, which is considered as uniform in calculating the equivalent air capacitance. A series of tests was made on specimens of different thickness varying the spacing between the guard ring and the main electrode from 0.025 to 0.2 in. The dielectric constant and the power factor were calculated for each spacing and specimen and the results plotted as a function of guard spacing. As the spacing increased, there was a falling off in dielectric constant as calculated which was more rapid the thinner the specimen. There was also a slight change in the calculated power factor. These curves were extrapolated to zero guard spacing and the resulting dielectric constant and power factor considered as the true constants of the material.

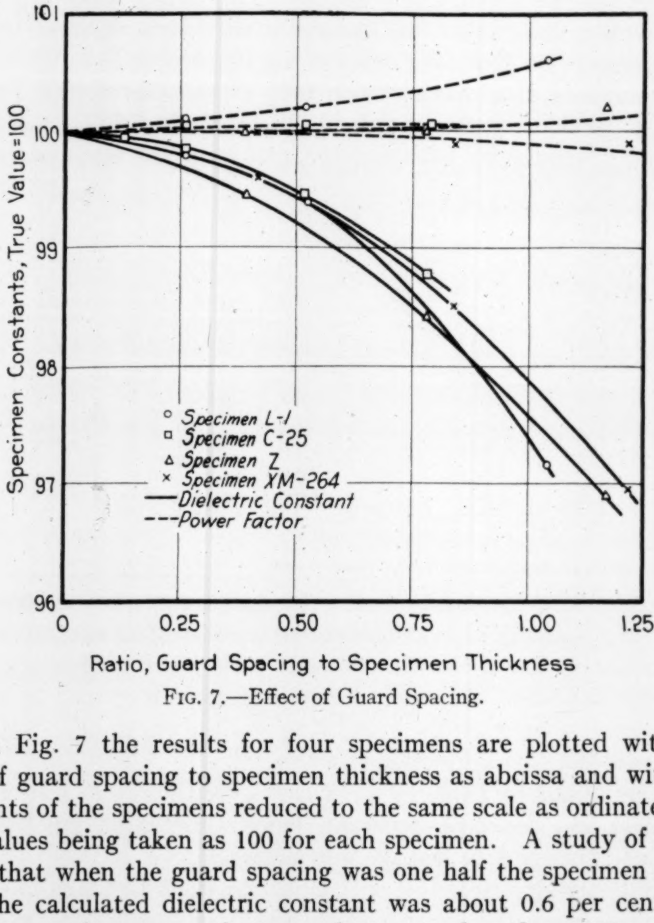

FIG. 7.—Effect of Guard Spacing.

In Fig. 7 the results for four specimens are plotted with the ratio of guard spacing to specimen thickness as abcissa and with the constants of the specimens reduced to the same scale as ordinates, the true values being taken as 100 for each specimen. A study of Fig. 7 shows that when the guard spacing was one half the specimen thickness, the calculated dielectric constant was about 0.6 per cent low, while the maximum error in power factor was less than 0.25 per cent for any of the four specimens. When the guard spacing was one-fourth of the thickness, the error in dielectric constant was 0.25 per cent and negligible in power factor.

The A.S.T.M. Standards call for a minimum specimen thickness of 0.1 in. This means that the guard ring spacing should be 0.025 in., which can be readily obtained provided the lead foil is cut with a pair of dividers.

Using the guard electrodes properly spaced or equal electrodes accurately centered gave good results at 1000 cycles. Experience indicated that it was somewhat simpler to use guard electrodes than accurately to center equal electrodes.

REPRODUCIBILITY OF RESULTS

There are so many variables and sources of error that enter into the measurement of dielectric constant and power factor of specimens that it is interesting to determine to what extent results are reproducible.

TABLE V.—MEASUREMENTS MADE IN THE SCHERING BRIDGE.

SPECIMEN	DATE	HOURS AT 30 C.	POWER FACTOR	DIELECTRIC CONSTANT
L-1	12/7/32	23	0.0368	5.93
	12/16/32	22	0.0368	5.93
	3/2/33	144	0.0369	5.90
L-2	12/1/32	21	0.0367	5.94
X-150-2	2/1/33	48	0.02005	4.73
	2/8/33	226	0.02005	4.74
	3/27/33	18	0.01975	4.73

The results of a number of measurements made at different times on various specimens in the Schering bridge are given in Table V. Specimen L-2 was cut from the same sheet as specimen L-1. From a study of the data in Table V and the results of other tests it was found that the results were reproducible to within 1 per cent in dielectric constant and to better than 5 per cent in power factor.

SUMMARY

1. The use of the substitution method of measurement eliminates inherent bridge errors.

2. The results obtained with solidly grounded bridges as recommended by the A.S.T.M. method are subject to errors due mainly to stray capacitances to ground.

3. The use of the Wagner ground eliminates errors arising from stray capacitances to ground, and permits guard electrodes to be used without complications in the bridge circuit. Means, however, should be provided for readily disconnecting the Wagner ground.

4. The series resistance bridge has ample sensitivity, but as a grounded bridge its accuracy is impaired by stray capacitances. A double shielded series resistance bridge allows the adoption of a Wagner ground and gives excellent accuracy.

5. The sensitivity of the parallel resistance bridge at 1000 cycles is increased by the use of larger resistances in the R_1 and R_2 arms. This is especially desirable when the bridge is used with small specimens of low power factor.

6. The balanced Schering bridge equipped with a Wagner ground provides in a single bridge a wide range of frequency operation, and would serve equally well for measurements at both 60 and 1000 cycles. It has ample sensitivity and accuracy.

7. Guard electrodes may be readily applied to specimens when using bridges employing the Wagner ground. With the proper guard spacing they eliminate edge corrections, and do not require a centering operation.

8. Equal centered electrodes, using the proper edge corrections, give results consistent with those obtained with guard electrodes. The edge corrections proposed by the Society were checked very closely considering the wide differences in specimens and conditions. The effect of surface leakage at 1000 cycles and 60 per cent relative humidity was found to be negligible.

9. It is important that specimens be maintained at constant temperature while testing until equilibrium is reached and that their constants should be measured throughout the working range of temperature.

RECOMMENDATIONS

The following practices are recommended for the measurement of dielectric constant and power factor of specimens at 1000 cycles:

1. The use of the substitution method.
2. The use of a Wagner grounded bridge provided with means for readily disconnecting the ground if desired.
3. The use of the balanced Schering bridge, equipped with Wagner ground, as the standard test method at both 60 and 1000 cycles.

REFERENCES

(1) Tentative Method of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (D 150–32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 820 (1932); also 1933 Book of A.S.T.M. Tentative Standards, p. 778.

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(7) G. M. L. Sommerman, to be published later.

(8) W. B. Kouwenhoven and A. Banos, Jr., "A High Sensitivity Power Factor Bridge," *Transactions*, Am. Inst. Electrical Engrs., Vol. 51, p. 202 (1932).

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(11) L. Hartshorn, "Dielectric Properties of Varnished Cloth at Low Voltage Gradients," *Journal*, Inst. Electrical Engrs. (London), Vol. 70, p. 417 (1932).

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DISCUSSION

MR. J. G. FERGUSON[1] (*presented in written form*).—At the time the tentative test for dielectric constant and power factor at radio frequencies was prepared, the subject of the various errors involved in the determination of dielectric constant and power factor was investigated thoroughly and a full discussion of these errors is to be found in the paper by Hoch[2] from which the fringe effect correction contained in the present method was taken. At that time the accuracy which was considered satisfactory at radio frequencies was such that correction for capacitance to ground from the high terminal of the sample was considered unnecessary. Later when the standard for 1000-cycle tests was written, this correction was again neglected although at this frequency the accuracy expected is high enough to make it appreciable, and enclosing the sample in a container for the purpose of maintaining control of temperature or humidity increases the correction.

This error should not perhaps be considered strictly a bridge error. For the computation of the dielectric constant, the capacitance required is the direct capacitance between the electrodes and the error consists in measuring the grounded capacitance. Any method which measures grounded capacitance will be equally in error. If the error is large enough to require consideration, some method should be used which furnishes a direct capacitance measurement. Such a measurement can be made using the parallel bridge, Fig. 3 of the paper, if modified by putting the double shielded transformer across *BD*, grounding the *C* corner and double shielding the adjustable standards. The measurement is then made by transferring the direct capacitance from the *CD* arm to the *AD* arm, as described by Campbell.[3] A more common method is the use of the Wagner ground which was developed by Wagner primarily to measure the direct capacitance of cable. This modification can be made readily to any of the bridges considered in the paper. It is unfortunate perhaps that the authors chose the series bridge to modify with the Wagner ground rather than the parallel bridge since a series bridge always requires more shielding.

The principal objections to the Wagner ground are that it requires additional controls and four balances are required for any measurement, two bridge balances and two Wagner ground balances.

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[1] Bell Telephone Laboratories, Inc., New York City.

[2] E. T. Hoch, "Electrode Effects on the Measurement of Power Factor and Dielectric Constant of Sheet Insulating Materials," *Bell System Technical Journal*, Vol. 5, p. 555 (1926).

[3] G. A. Campbell, "Measurement of Direct Capacitance," *Bell System Technical Journal*, Vol. 1, p. 18 (1922).

The substitution method is undoubtedly the most accurate method of operation, the only objection to it being that it requires a little additional equipment. For accurate results this method is essential in the case of the Schering and series resistance bridges, but not in the case of the parallel bridge. It can be avoided by making the original calibration by substitution and using the direct comparison method for regular operation if the bridge is adequately shielded.

From the operator's standpoint, the bridge should be as nearly direct reading as possible. While none of these bridges measures power factor directly, and in fact it would be a very difficult matter to design one to do so, on the other hand the parallel bridge is practically direct reading for conductance while the other bridges do not read directly either conductance or resistance.

Regarding sensitivity, it is obvious that for the particular purpose considered where the reactance of the sample is in the order of 500,000 ohms, all impedances should be as high as practicable, including the impedances of the source and detector. It is somewhat questionable, however, how far we should go in this direction if it involves a sacrifice of the general usefulness of the bridge for other purposes, since sensitivity is usually rather a minor consideration.

In conclusion I would recommend the following changes in Tentative Method D 150 - 32 T:[1]

For 1000-Cycle Tests:

1. Omission of the bridge of Rosa and Grover.
2. Modification of the series resistance bridge to include a Wagner ground.
3. Inclusion of the parallel resistance bridge modified both by the use of the Wagner ground and by shielding adequately to permit the measurement of direct capacitance without the Wagner ground.
4. Inclusion of the Schering bridge using a Wagner ground and the substitution method.

For 60-Cycle Tests:

1. Modification of the Schering bridge to include a Wagner ground.
2. Modification of the parallel resistance bridge either by including a Wagner ground or by shielding and grounding it so that it will measure direct capacitance without the Wagner ground.

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[1] Tentative Method of Testing Electrical Insulating Materials for Power Factor and Dielectric Constant (D 150 - 32 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, p. 820 (1932); also 1933 Book of A.S.T.M. Tentative Standards, p. 778.

MR. I. M. STEIN[1] (*presented in written form*).—The authors recommend (1) the use of the substitution method, (2) the use of a Wagner grounded bridge provided with means for readily disconnecting the ground if desired, and (3) the use of the balanced Schering bridge, equipped with Wagner ground, as the standard test method at both 60 and 1000 cycles.

The first recommendation would appear to be unwarranted, although there is no reason why a substitution method should not be used if its use does not involve complications in the balancing of the bridge. In general, substitution methods require extra operations, but these extra operations cannot be avoided when the accuracy sought in the result is beyond the limit that can be obtained with direct-reading apparatus. In some cases, even though adequate direct-reading apparatus is available generally, it is not available to the particular user and he must resort to substitution methods. Then too, even though the direct reading apparatus were available to the particular user, he might be in some doubt with regard to its calibration at the time. In this case, however, a single substitution measurement or some other check provided in the apparatus usually would settle the question as to whether or not the apparatus was in proper calibration.

Considering the requirements to be met by the apparatus as specified in the tentative methods, it seems clear that direct-reading apparatus could be obtained from several sources to meet the requirements.

It should be pointed out that the apparatus shown in Fig. 4 of the paper, which shows the recommended balanced Schering bridge, does not utilize a true substitution method, but only a partial substitution method. The method is a substitution one in so far as the capacitance measurement is involved, but the more important measurement of power factor requires the operation of condenser C_1, which is not in the "X" arm of the bridge.

Referring to the second recommendation, we are in agreement that the Wagner ground connection be specified as one satisfactory method of avoiding lead errors in the measurements called for by the tentative method. The ratio box, which was used in some of the measurements discussed in the paper, is, as the paper states, regularly equipped with a Wagner ground connection. Simple means for disconnecting the ground can readily be provided, but it should be pointed out that no such means are indicated in Fig. 4 of the paper, showing the balanced Schering bridge.

In agreeing that a Wagner ground may be used on the bridge, we do not agree with using it in the manner shown in the balanced Schering bridge of Fig. 4. A well-known objection to the ordinary Wagner ground connection, as shown in Fig. 4, is that it complicates the operation of the bridge because the Wagner ground circuit has to be readjusted each time the main bridge setting is changed and a trial and error method is necessary to obtain a simultaneous balance in the main bridge and in the Wagner ground circuit. Such successive adjustment of the Wagner ground would appear to be necessary in making the initial balance on the balanced Schering bridge and again when making the final balance due to the fact that the final balance is obtained by adjusting condenser C_1. With a fixed 1 to 1 ratio arm bridge as used in the parallel resistance bridge indicated in Fig. 3 of the paper, the initial adjustment of the Wagner ground can be made very conveniently in a direct manner and the Wagner ground balance is then fixed for all later adjustments of the main bridge.

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Also in agreeing that a Wagner ground be specified as one satisfactory method for taking care of lead errors, we do not agree that the Wagner ground be specified as the only satisfactory method for the purpose. As is well known, the solidly grounded bridge may be used for the measurement of direct capacitances, and when so used, the lead capacitances do not enter into the measurement. For instance, if we consider the sample holder with its shielded leads as a direct capacitance which it is desired to measure and having two auxiliary capacitances, one in one lead and one in the other, and consider a ground point bridge as shown in Fig. 3 of the paper, then the direct capacitance in the sample holder can be determined from two bridge balances as follows: In the first bridge balance, the direct capacitance is across the points *DC*, one of the auxiliary capacitances is across the points *AD*, and the other auxiliary capacitance is across the points *AC*. For the second balance, the two conductors connected to point *C* are disconnected from point *C* and connected to point *A*. For this connection the direct capacitance is placed in parallel with the auxiliary capacitance which was across the points *AD* in the first balance and the auxiliary capacitance which was across the points *AC* is short circuited. The direct capacitance is found by taking one-half the difference between the two bridge balances. With connections properly provided in the bridge, the switching of connections between the first balance and the second balance can be made without introducing any error due to auxiliary lead capacitances. This method is more appropriate than the Wagner ground method when making measurements under such conditions that one terminal of the direct capacitance must be grounded at all times. The Wagner ground method is the appropriate method when measurements are to be made under such conditions that neither terminal of the direct capacitance is grounded, the ground being on the enclosing shield.

Referring to the third recommendation, while it might be desirable to include the balanced Schering bridge equipped with Wagner ground as one acceptable method, the recommendation that it be adopted as *the* standard test method at 1000 cycles seems unwarranted.

The balanced Schering bridge as shown in Fig. 4 has a singly shielded input transformer whereas we should recommend the use of a doubly shielded transformer in order to avoid inconvenience due to variable conductance in the transformer itself. The commercial parallel resistance bridge described in the paper is provided with a doubly shielded transformer for this reason.

The parallel resistance bridge described in the paper is not only useful for measurements at 1000 cycles, but as regularly made is useful up to 50,000 cycles, and by merely changing the input transformer to one with more suitable characteristics may be used at frequencies up to radio frequencies.

Referring to the precision of setting with the 10,000-ohm resistors for R_1 and R_2, R_1 being adjustable in steps of 0.01 ohm, and considering a test specimen with a capacitance of approximately 500 $\mu\mu$f. as used in the measurements described on page **831** in the paper, a variation in one step on the 0.01 ohm dial of R_1 corresponds to 0.00003 in power factor at 1000 cycles. This corresponds to 3 in the last decimal place in the measurements discussed in the paper, whereas for the requirements of the tentative methods, a precision of setting of 3 in the next to the last decimal place would be adequate for all of the measurements indicated in the paper. Accordingly, even when used with the low sensitivity detector used by Kouwenhoven and Marks, the parallel resistance bridge is capable of measurements well beyond the accuracy and precision called for by the tentative methods. According to our understanding of the amplifier used by Kouwenhoven and Marks, it has a low amplification ratio (in the order of 20). Using the relatively inexpensive oscillator and tuned detector amplifier intended for use with this bridge, a sensitivity considerably greater than would be necessary to read to the last decimal place shown in the paper is available.

Referring to accuracy in the parallel resistance bridge, the only limitation in the power factor measurement is the phase defect in the ratio coils, and the residual inductance or capacitance tolerance in these ratio coils is so small that at 1000 cycles, the maximum error in power factor from this cause would not exceed 0.000001. This is so small as to be altogether negligible.

It would seem from some of the discussion in the paper that the authors have in mind a type of bridge useful for measuring low-power-factor oil samples rather than solid dielectrics, as called for in the tentative methods. While it would appear to be highly desirable to adopt standard test methods for solid material which could be readily modified when the power factor of oils is to be measured, it would seem to us to be very unwise to burden the purchaser of an equipment for measuring solid materials with the added cost of the equipment necessary for measuring low-power-factor oils. With the parallel resistance bridge, the precision of setting necessary for measurements on low-power-factor oils can be obtained quite simply by adding two units to the bridge—one a fixed 90,000-ohm resistor to be connected in series with the variable resistor R_1, and the other a 100,000-ohm fixed resistor to be substituted for R_2. With this change, the precision of setting would be 100 times as great as when the 10,000-ohm resistors are used and there would also be a corresponding gain in sensitivity which would be useful in measurements on low-power-factor oils.

The balanced Schering bridge of Fig. 4 appears to be open to another objection in addition to the objection already cited of inconvenience in balancing the Wagner ground. This has to do with its use at comparatively high voltage. The Schering bridge has been given a great deal of publicity since it was first proposed in 1920. The publicity has resulted in rather extensive use and the extensive use has led to refinements which have made it possible to construct really precise bridges of this type. However, the Schering bridge has no particular merit with regard to precision, as completely shielded bridges capable of extreme precision were described by Campbell in 1904, have been improved and extended by Shackelton, Ferguson and others, and have been used extensively. The real point of merit in the Schering bridge is that when used for high voltage measurements, the adjustments to balance the bridge can be made on units which are very nearly at ground potential. This leads up to the point that the balanced Schering bridge shown in Fig. 4 of the paper sacrifices this one outstanding point of merit in the Schering bridge, as some of the adjustable units in the Fig. 4 bridge are at high voltage when high voltages are used. This does not mean to say that the Fig. 4 bridge is not an acceptable method for making the measurements called for by Methods D 150 – 32 T, but is intended merely to indicate that the bridge shown has no particular merit over precise bridges of other types.

MR. DEAN HARVEY[1] (*by letter*).—I wish to call attention to a portable power factor instrument recently placed on the market, primarily for testing the insulation of transformer and circuit breaker bushings in the field. The power factor set uses the inverted Schering bridge principle, arranged to allow the testing of bushings. The novel feature of this portable bridge is that bushings can be tested without having to drop the oil in the tank to disconnect the bushing from the winding. Tests have been made successfully on bushings in the field ranging from 6.9 to 230 kv.

Power factor and capacity tests have given a more complete knowledge of the condition of bushing insulation than any other tests. The value of a test that can predict trouble before failure actually occurs cannot be overestimated.

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[1] Materials Engineer, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

An article describing the design and tests of this bridge was published in the January, 1934, issue of *Electrical Engineering* under the subject of "Portable Schering Bridge for Field Tests," by Messrs. Watts, Burr and Hill.

MESSRS. W. B. KOUWENHOVEN[1] AND L. W. MARKS[2] (*authors' closure by letter*).—Perhaps the circuit diagrams in our paper give the impression that we do not recommend the use of the Wagner ground with the parallel resistance bridge. Such, however, is not the case as we stated in the text, page 826, that the Wagner ground may be used with the parallel resistance bridge.

Mr. Ferguson in his discussion has presented a clear picture of the importance of ground capacitance. Unless it is either eliminated or corrected for, its presence will cause an error not only in the calculated dielectric constant but also in the power factor of the specimen.

The Campbell method of measuring direct capacitance does not allow the simultaneous measurement of the true power factor.

Mr. Stein is correct in his statement that the bridge shown in our paper does not use a strict substitution method. In the text, page 829, we recognized this and named it a "parallel substitution method." This particular type of substitution has several advantages. It is also just as accurate for power factor measurements as for capacitance.

We recommend definitely that all bridges equipped with Wagner grounds should be provided with means for readily disconnecting same. We indicated by means of double dots (Figs. 2 and 4) that the Wagner ground was connected to binding posts at these points.

We had no difficulty in adjusting the Wagner ground in any of the three bridges studied. It must be kept in mind that when the specimen is connected in the parallel resistance bridge, its capacitance to ground affects the Wagner ground balance, which must be readjusted to take care of the change.

As pointed out by Mr. Stein, there are other methods than that of the Wagner ground for eliminating lead errors. For example, in our high-voltage cable measurements at Johns Hopkins University we use solidly grounded bridges[3] in which such errors are eliminated. In these solidly grounded bridges doubly shielded transformers are used to connect the detector to the bridge. At low voltages, however, a Wagner ground simplifies the measurements.

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[1] Professor of Electrical Engineering, Johns Hopkins University, Baltimore, Md.

[2] Student Engineer, General Electric Co., Schenectady, N. Y.

[3] W. B. Kouwenhoven and A. Banos, Jr., "A High Sensitivity Power Factor Bridge," *Transactions*, Am. Inst. Electrical Engrs., Vol. 51, p. 202 (1932).

THE VIBRATING WIRE METHOD OF MEASURING DEFORMATIONS

BY N. DAVIDENKOFF[1]

The enormous construction program being carried out by the Union of Soviet Socialist Republics presented the problem of verifying under natural circumstances the technical data forming the basis of these engineering projects. This work required an instrument for use in tension, pressure, deformation, settling, etc. This led the Institute of Technical Physics and the Institute of Buildings at Leningrad, in 1926, to start the development of the vibrating wire method for measuring deformations in engineering structures. The development was carried out simultaneous to but independent of Schaefer's work **(1)**[2] on the same problem in Germany.

THE PRINCIPLE OF THE WIRE METHOD

The method depends upon the measurement of the pitch or vibration of a stretched wire, which in turn is a measure of the tension applied. The fundamental natural frequency of a stretched wire is:

$$N = \frac{1}{2l}\sqrt{\frac{\sigma}{\rho}} \quad \text{(1)}$$

where N = the fundamental frequency (pitch of the emitted sound),
l = the length of the wire between stretching grips,
σ = tensile stress in the wire, and
ρ = density of wire material (mass per unit volume).

When the distance between the points of support is changed slightly, the stress in the wire changes rapidly, giving rise to a change in the natural frequency of the wire. From Eq. 1:

$$\Delta\sigma = 8l^2\rho N\Delta N \quad \text{(2)}$$

The accuracy of the vibrating wire method of measurement depends largely on the accuracy of measuring the vibration frequency of the wire. In the field the frequency can be determined with an error of 1 to 2 cycles per sec. In the laboratory the error is reduced to 0.1 cycle per sec.

From Eqs. 1 and 2:

$$\Delta N = \frac{\Delta\sigma}{4l\sqrt{\rho\sigma}} \quad \text{(3)}$$

For a given $\Delta\sigma$, the sensitivity, ΔN, is increased by decreasing l and σ.

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[1] Professor of Testing and Strength of Materials, Industrial Institute, Institute of Technical Physics, Institute of Buildings, Leningrad, U. S. S. R. (Russia).

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 860.

The lower limit for σ is reached when the wire no longer emits a pure tone owing to its own stiffness(2). A stress less than 15 kg. per sq. mm. is not usable. Similarly if l is too short, end conditions become too important. For a diameter of 0.2 mm. the length should be at least 5 cm. Lengths greater than 6 cm. are preferable.

For $\Delta N = 1$ cycle per sec., $\sigma = 15$ kg. per sq. mm.; $\rho = 0.000008$ kg. per cu. cm. (steel wire) and $l = 4$ cm., $\Delta\sigma = 1.75$ kg. per sq. cm. corresponding to an extension of 0.000035 mm. or a strain of about 0.000001 cm. per centimeter. If $l = 10$ cm. and $\sigma = 40$ kg. per sq. mm., 1 cycle per sec. change in frequency corresponds to a change in stress of 7.2 kg. per sq. cm. or a strain of 0.0000035 cm. per centimeter.

In practice the error in measuring strains does not exceed 0.000010 cm. per centimeter. In cases where the gage length is longer than the vibrating wire the error is reduced (for example, in the tensometer the error does not exceed 0.000005 cm. per centimeter).

Naturally the actual relationship between ΔN and $\Delta\sigma$ is obtained by calibration and not by using Eqs. 1, 2 and 3.

In cases where the measurements of actual deflections are important (earth dynamometers, expansometers) Eq. 3 can be written:

$$\Delta N = \frac{E\Delta l}{4l^2\sqrt{\rho\sigma}}$$

where E is the modulus of elasticity of the wire material. From this it is seen that it is very important to use small values of l.

METHOD OF MEASURING THE PITCH OF THE WIRE NOTE

The method of measuring the pitch of the extensometer vibrating wire was the subject of a long development. The first method tried was a comparison method in which a calibrated, tuneable wire was stretched until its frequency coincided with the frequency of the extensometer. In order to insure accuracy and the elimination of personal error several electrical systems were developed. In all these systems an electromagnet placed near the wire acted as an exciter and in the final development also as a detector. Three general electromagnetic methods were tried:

1. Resonance method.
2. Self-excitation method.
3. Method of listening to damped oscillations.

Certain disadvantages in the self-excitation method and the method of listening to damped oscillations eliminated them from consideration and the resonance method was chosen as the most suitable for continued use.

In this system an electromagnet with windings fed from an audiofrequency triode oscillator with suitable amplification is placed near the center of the extensometer wire which must be made from a magnetic material. The oscillator is tuned until the frequency of the magnetic pull is in resonance with the lowest natural frequency of the wire. When the wire is in resonance, the sound it emits becomes intense enough to be heard by the ear or through a microphone. In places where it is impossible to use direct observation by ear or by microphone a second electromagnet can be used as a detector of the vibration. In the final stage of the development the same electro-

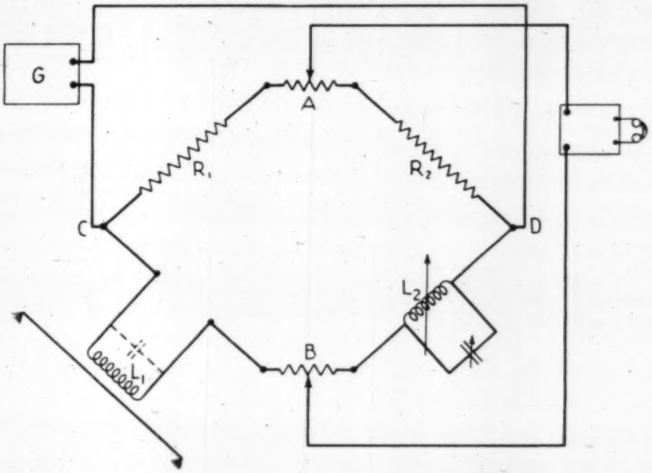

FIG. 1.—Essential Features of Method of Measuring Pitch of Wire.

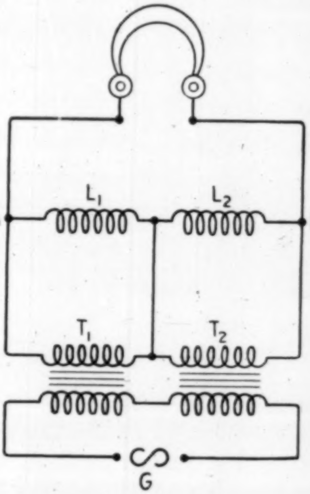

FIG. 2.—Variant of the Bridge Hook-up.

magnet acted both as an exciter and a detector, thereby eliminating the double transmission needed for the method using two magnets.

The essential features of this system are shown in Fig. 1. The electromagnet is placed near the wire and its winding L_1 is connected as a self-induction in one arm of a bridge. The other arms consist of the self-inductance L_2 and two resistances R_1 and R_2. The current of the generator is applied at C and D. When $L_1 = L_2$ and $R_1 = R_2$ no current flows in the diagonal AB. If the wire vibrates, L_1 changes, unbalancing the bridge, and an alternating current flows through A and B to the amplifiers connected to the observer's telephone. Figure 2 shows a variant of the bridge hook-up which was adopted as the final form to be used. Two transformers T_1 and T_2 supplied by the same generator oscillate with equal amplitude and frequency, but in opposite phase. One of the arms of the bridge contains the self-inductance L_1 of the electromagnet near the vibrating wire and the other arm contains a balancing self-inductance L_2. When the wire begins to resonate the balance of the bridge is disturbed and a signal is heard in the telephone. Figure 3 shows the diagram of the

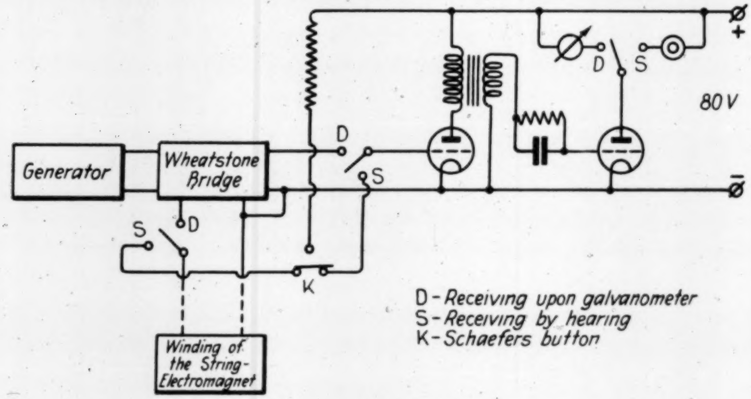
Generator
Wheatstone Bridge
D
S
K
Winding of the String-Electromagnet
80 V
+
−
D - Receiving upon galvanometer
S - Receiving by hearing
K - Schaefers button

FIG. 3.—Diagram of Complete Hook-up.

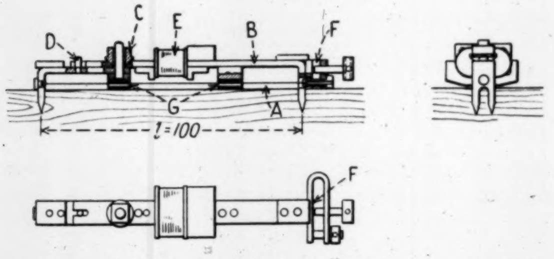
C
E
B
F
D
G
A
l = 100
F

FIG. 4.—Extensometer Suitable for Measuring Deformations in Wooden Beams.

complete hook-up used. The signals can be received aurally by closing the contacts S or visually by closing the contacts D.

VARIOUS APPLICATIONS OF METHOD

The methods and instruments described above have been used on a great variety of civil engineering projects and on some mechanical problems as described in detail below.

Measurement of Stresses in Structures:

Figure 4 shows an extensometer suitable for measuring deformations in wooden beams. The wire, A, is fixed to a removable steel frame, B, and held by a conical clamp, C, and guide pin, D. The distance of the wire from the beam under investigation is kept constant by a stretcher, G, with a rubber washer. A spring clamp, F, is used for tuning. The instrument is fixed to the wood by means of sharp nails. Naturally other suitable clamps could be used

Experimental measurements of rock pressures in tunnels have been made by measuring the stresses in the upper and side props in mine sets of the trapezoidal type. The extensometer is attached near the point of maximum bending moment and the set is unloaded by removing the load from the trapezoidal frame as by means of temporary auxiliary supports. As many as ten consecutive sets are measured in order to obtain a mean value along the tunnel. From the stress in the caps the load on each square meter of the roof can be calculated by assuming either a uniform or parabolic load distribution.

In order to calculate stresses from the measured strains, the modulus of elasticity of the wood must be measured. For this purpose each prop with its extensometer was removed to the surface and tested as a simple beam by loading with a pan and weights. The direct relationship between bending moment and strain secured in this way eliminates possibilities of large errors. A mean deviation of 16 per cent in the value of Young's modulus of elasticity has been obtained at Ulbastroi. The table below gives some values obtained at the various projects named:

Name of Construction	Type of Rock	State of Strata	Number of Frames	Mean Span, m.	Load, tons per sq. m.	Remarks
Zoragetstroi[a] (1929) (**4**)	Dacite	Strongly weathered	1	2.0	7.0	On the assumption of uniform load distribution.
		Less weathered	5	2.0	2.1	
Gizeldonstroi[a] (1930) (**5**)	Limestone	Very friable	5	1.75	7.0	
Ulbastroi[b] (1931)	Epidite-chlorite-quartz shale	Strong	42	1.96	0.25 to 0.68	On the assumption of parabolic distribution.
		Weak		1.96	0.94	
		Disintegrated		1.96	1.45	
Daljeldorstroi[c] (1932)	Kaolinized tuffs	Moist	7	2.27 to 2.70	2.55 to 5.1	
		Saturated	4	2.30 to 2.37	6.45 to 8.45	
	Weathered tuffs	Tough	3	4.55 to 4.65	0.83 to 1.18	
Metrostroi[d] (1933) (**6**)	Jurassic	Moist	5	1.53 to 1.64	3.0 to 11.9	
			2	6 to 7	42.0	

[a] Caucasas. [b] Altai. [c] Siberia. [d] Moscow.

Measurements of Stress Inside Concrete:

The difficulties in measuring stresses inside concrete arise from the inaccessibility of the point of observation and the large volume changes which take place during a protracted experiment as a result of chemical processes accompanying setting and aging, swelling and shrinking, together with changes in the modulus of elasticity as the concrete ages.

The essential characteristic of a teletensometer for concrete is that it shall not distort the field of stress acting on it and accordingly it must have a longitudinal rigidity much less than the rigidity of the concrete. It must, however, be rigid enough to maintain the vibrating wire at its original pitch while concrete is being poured around it. Figure 5 shows an instrument satisfying these conditions. The body of the instrument is made of thin brass tubing, *A*, with a deep flexible neck located near wire socket, *B*. Two cross pieces, *C*, are fixed at the ends of the instrument to secure a good connection with the concrete. The wire is held by a pair of split cones, *D*, seated in sockets, *B*, and *E*. The heads, *E*, are joined to the cross pieces by temperature compensating joints using the difference between the coefficients of thermal expansion of brass, *E*, and steel, *F*, and designed to compensate for the difference between the expansion coefficients of the steel wire and the surrounding concrete. The tube is covered with coal tar pitch which protects it from water and keeps it from sticking to the concrete. It is essential to keep the tube neck free from all mastic. The length of the wire is 100 mm., the gage length (distance between cross pieces) is 200 mm. which doubles the sensitivity of the instrument.

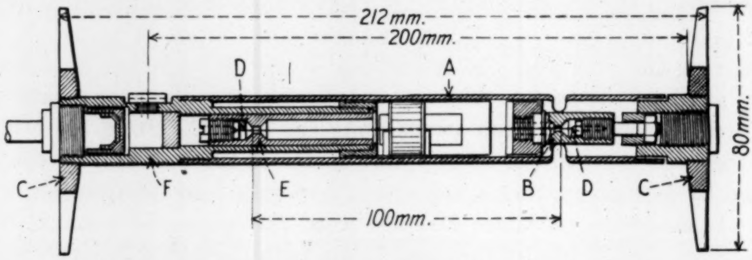

FIG. 5.—Teletensometer for Concrete.

In fixing the instruments in place, care must be exercised in the ramming process to see that they are not displaced or distorted. A sufficient number of instruments properly oriented must be used. For the measurement of a three-dimensional stress six tensometers must be placed at (near) each test point; three along mutually perpendicular axes and three along the bisectors of the angles formed between the mutually perpendicular instruments. Where one of the principal axes is known because of symmetry in the structure, four instruments can be used; if all three principal axes are known, three instruments lying along these axes are sufficient.

It is important to distinguish between changes due to chemical or physical processes and those due to mechanical loading. Unfortunately it is almost impossible to make an accurate distinction between these effects. The tensometers should preferably be used only after the setting of the concrete is complete. For extended observations the most reliable method is to have a volume of concrete under the same conditions of temperature, humidity, etc., but isolated from the influence of mechanical stresses. Controlled tensometers are distributed in this volume of concrete and their readings are subtracted from those of the working instruments.

In order to investigate the reliability of the tensometer, an instrument was sealed in an 18-cm. cube of concrete. Two Marten's mirror extensometers with 150-cm. gage length and optical levers 10 m. long were attached externally to the block. The average of 35 readings taken under loads ranging from 1500 to 4000 kg. (stresses in the concrete from 4.6 kg. per sq. cm. to 12.3 kg. per sq. cm.) with the vibrating wire agreed within 0.7 per cent with the mean of the readings of the Marten's extensometers. The mean deviation of the individual readings from the average values was 2.3 per cent.

In 1931 at Zoragetstroi(7), tensometrs were placed in two cross-sections in the concrete facing of a pressure tunnel 3 m. in internal diameter. Eight instruments were fixed in each cross-section at the ends of the vertical and horizontal diameters near the inner and outer surfaces of the facing. The test section of the tunnel was isolated by partitions through which the instruments leads were passed. The section was filled with water and the pressure was supplied and released twice. Thirty readings on each instrument were taken; but only those taken during the last reduction of pressure were consistent. This may be explained upon the assumption that the water gradually penetrated the concrete and disturbed its homogeneity. The following table shows a comparison of the mean value of the deformations as measured by the extensometers and as calculated theoretically from the elastic constants of concrete of this type:

	DEFORMATION	
	MEASURED	CALCULATED
Tunnel section I	0.0000118	0.0000122
Tunnel section II	0.0000089	0.0000091

Individual readings of the tensometers had a mean deviation of 35 and 25 per cent.

Professor U. Neelander made measurements with 204 tensometers in the dam at Dneprostroi(8). The tensometers were distributed in groups of six, one vertical, one horizontal, and two at an angle of 45 deg. in a plane parallel to the face of the dam, one parallel to the axis of the dam, and one for control in a volume isolated from the stress conditions. Above each group a telethermometer was placed. This instrument, similar to the tensometer, has no thermal compensation and is not connected mechanically to the concrete. The telethermometer used at Dneprostroi could be read to 0.1 C.

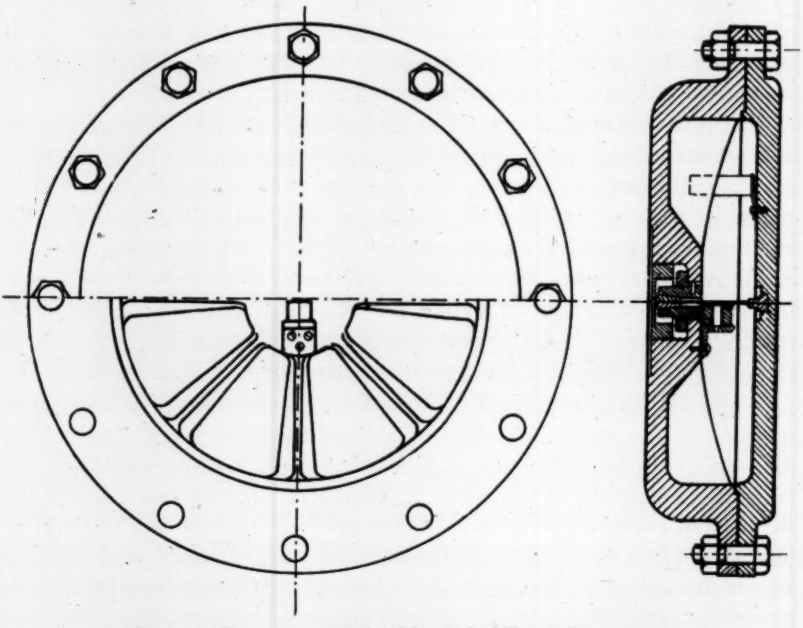

FIG. 6.—Dynamometer for Measuring Earth Pressures.

These investigations show, contrary to common belief, that large bodies of concrete do not shrink while setting. In small laboratory models where the concrete cools quickly, surface drying does bring about shrinkage of volume; in large bodies the surface plays a secondary role and temperature effects predominate.

Temperature measurements on the dam at Dneprostroi showed that nine to ten hours after mixing the concrete, the rate of heating increased abruptly, indicating that setting had begun. After two to three hours the temperature continued to rise, though at a slower rate, reaching a maximum after one to five days (depending on the location of the instrument); the total rise was 17 to 20 C. Since heating continues after the setting of the cement, and since it is impossible for structures such as dams, which are constrained laterally, to expand parallel to the axis of the dam, a compressive stress is set up. These stresses were also measured with tensometers and found to be very considerable; reaching 30 to 40 kg. per sq. cm. while the stress calculated from the hydrostatic head should have been less than 12 kg. per sq. cm.

In the surface layers the rapid cooling and drying may give rise to tensile stresses of considerable magnitude, leading to the formation of cracks.

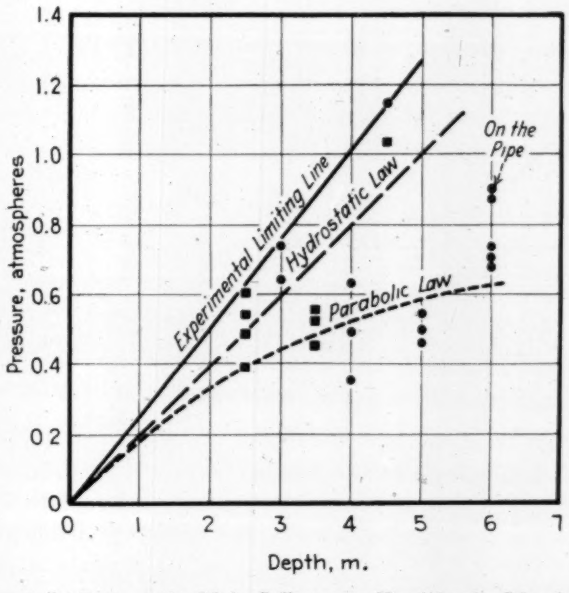

FIG. 7.—Results of Tests at the Main Collector on Vassielevsky Island, Leningrad.

Measurement of Earth Pressures(9):

Figure 6 represents a dynamometer for measuring earth pressures consisting of a flexible diaphragm or lid clamped to a comparatively rigid base. The earth pressure deforming the lid shortens the vibrating wire strung between the center of the lid and the center of the base. For an accuracy of 1 per cent the maximum deflection of the diaphragm must be limited to 0.01 mm.

Temperature changes affect the readings of the instruments because of different coefficients of linear expansion of the metals used in the wire and in the walls of the box, and due to changes in pressure of the air contained in the hermetically sealed boxes. In order to eliminate the temperature effects, a compensating device was constructed similar to that used in the teletensometer. The changes in air pressure are also compensated for.

Forty dynamometers were buried at various depths, up to 6 m., in different positions, vertically and horizontally, to measure the earth pressure on the new sewage system installed in Leningrad in 1928 and 1929. Figure 7 shows results of tests at two points in the section of the main collector on Vassielevsky Island (sandy soil, partly fluid). Calculated values based on the hydrostatic law are shown in the dashed lined and calculated values based on the usually assumed cubic parabola law are shown in the dotted line.

Measurement of Modulus of Elasticity of Rock Massifs:

In designing pressure tunnels for hydroelectric stations the problem of the stresses in the tunnel lining is statically indeterminate and it is necessary to know the modulus of elasticity and Poisson's ratio for the rock through which the tunnel is driven. Tests must be made in the field under natural conditions, since to attempt to make them in the laboratory on small specimens may eliminate effects of cleavage, cracks, inhomogeneities, etc. Measurements of this nature for the Zoragetstroi project (10) were carried out in a small mine shaft (1.5 m. in diameter) sunk near the site of the actual tunnel in the same strata. Air, instead of water, was used to apply pressure. The reduced scale of the experiment demanded measurements with an accuracy of 1 μ.

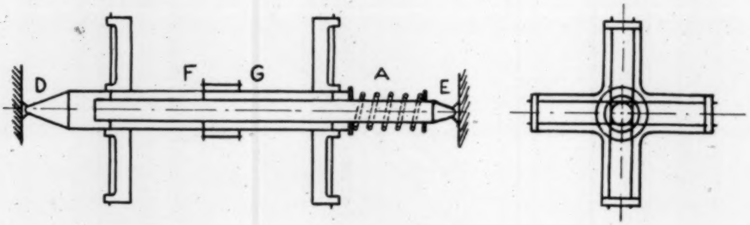

FIG. 8.—Expansometer.

The "expansometer" designed for this is shown in Fig. 8. It consists of two concentric steel tubes spread apart by the spring A and resting on points D and E against the walls of the shaft. The wire FG connects the tuning bridge in each tube and measures the displacement between the points D and E. The two tubes are kept exactly concentric, with no lateral play and no relative rotation, by suspending the inner tube on eight tangentially fixed thick strings. The strings are distributed in planes perpendicular to the axis of the tubes and attached to aluminum cross pieces fixed to the outer tube.

A free axial movement of ±1 cm. is possible with this device. The instruments are protected from temperature fluctuations by enclosing them in layers of insulating wadding.

The experimental shaft, 4 m. in depth and 1.5 m. in width, was faced with concrete 20 cm. thick and coated with tar to render it air-tight. It was sealed by a reinforced-concrete cover held by 16 bolts fitted to the bottom of the shaft. Air pressures up to 3 atmospheres were applied.

The deformations of the inner diameter, as measured by the instruments, varied from 30 to 50 μ, with very little scatter of the individual results. The elastic constants for the rock strata were calculated according to Lamé's formula. In applying this formula it is essential to know the elastic constants of the concrete used for facing the tunnel, to be determined by laboratory tests.

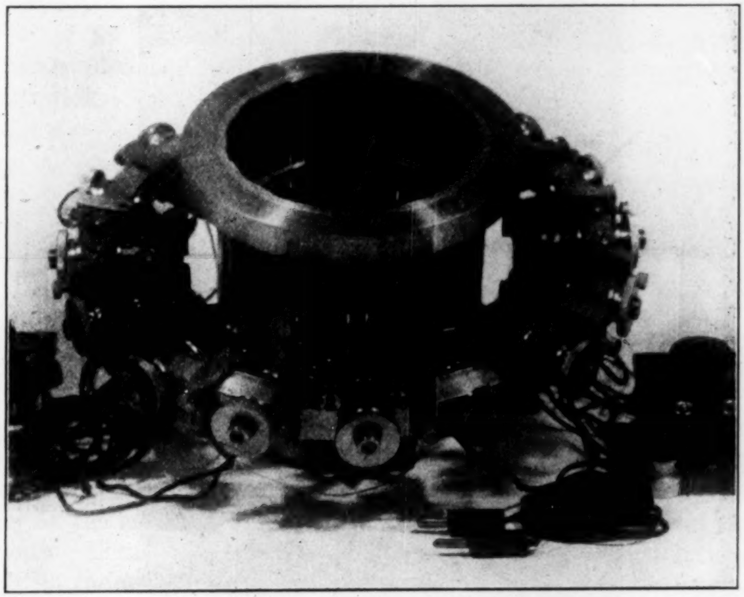

FIG. 9.—Instrument for Measuring Pressure Between Piston Rings and Cylinder Walls.

The values obtained made it possible to redesign the reinforcement of the reinforced concrete facing which has resulted in great economy in construction.

Special Applications:

An instrument for measuring pressure between piston rings and cylinder walls is shown in Fig. 9. It consists of a bored cylinder in the middle section of which narrow slits are cut. The vertical slats thus formed bend outward (not more than 0.005 mm.) under the pressure of the ring. This bending is measured by means of vibrating wires joining the centers of the slats to a special ring mounted on brackets. The deflection of the slats should not be large enough to diminish the ring pressure appreciably. To eliminate inaccuracy due to cylinder boring the measurements are made *n* times, rotating the ring through an angle of $\frac{2\pi}{n}$ between each measurement (n = the number of slits).

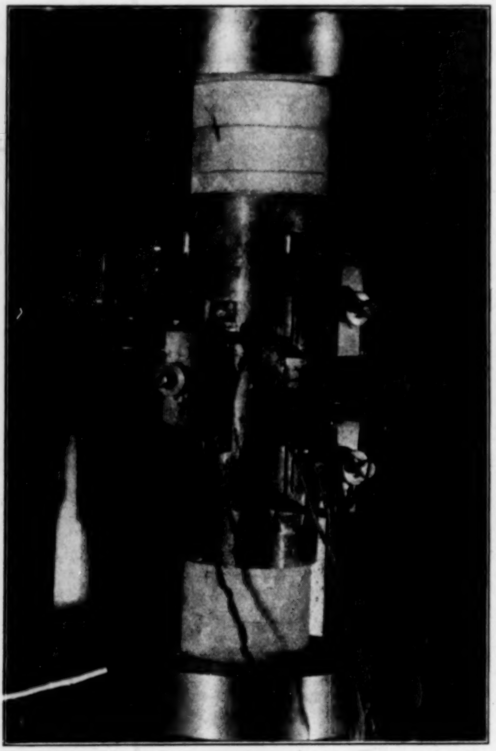

FIG. 10.—Instrument for Measuring Earth Thrusts.

An instrument for measuring earth thrusts is shown in Fig. 10 and consists of a cylinder made of thin brass containing a series of paired parallel slots. These slots divide the wall into narrow slats which bulge outward on the application of internal pressure. The cylinder is filled with earth contained in a thin rubber balloon and loaded by means of a wooden piston in a testing machine. The bending of the slats is measured by means of a vibrating wire extensometer and the instrument is calibrated by air or water pressure. An instrument of this type was tested at the Leningrad Research Institute for Hydrostatics and is being used at Metrostroi (Moscow) for studying the mechanical properties of soil.

Dynamical Measurements:

All the cases so far considered have dealt exclusively with static loading where the observer has time to measure the frequency of vibration. However, we can apply the method in principle to the study of rapid processes, provided the period of oscillation of the wire is sufficiently short (less than one tenth) in comparison with the period of the process being studied. For this purpose it is only necessary to change the measuring scheme and employ photographic registration.

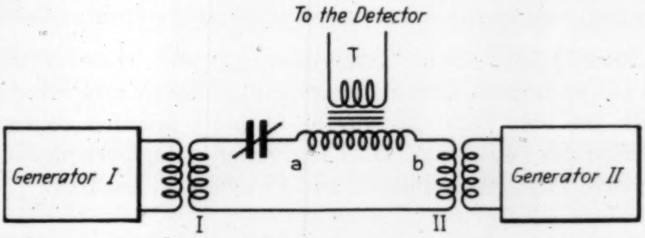

FIG. 11.—Self-Exciting Circuit.

A method was worked out with the physicist Lazareff in 1932 and applied in Zoragetstroi for measuring stresses in reinforced-concrete frame structures supporting the turbogenerators. The method is based on the use of the phenomena of forced synchronization of two oscillating circuits. One of the circuits (Fig. 11) is the self-exciting circuit of the wire, built for example according to the double magnet scheme; this circuit provides great power. The second generating circuit, on the contrary, takes very little power and is weakly coupled to the first by means of the coils I and II. If both generators are oscillating close to one another, the more powerful generator I will force its frequency on the weaker generator II. When this happens, both circuits oscillate with a certain phase difference depending on the degree of dissonance. As a result, a variable change of potential occurs between the points *a* and *b*. If now a periodic change of tension occurs in the element to which a wire has been stretched, the pitch of the wire will also vary periodically, which in its turn brings about a periodic change of dissonance (difference of frequency) in the circuits. It can be shown that the amplitude of the potential differences at *a* and *b* produced by these oscillations is directly proportional to the change of tension in the wire, provided this change is small.

The potential differences are transmitted by a transformer *T* to a detecting apparatus equipped with a Siemens oscillograph (characteristic frequency 100 hertz); the oscillations of the mirror are recorded on a photographic plate. The apparatus is calibrated by varying the pitch of the wire artificially and comparing the magnitude of the variation with that of the deflection of the spot of light. In making measurements with this method at Zoragetstroi, it was possible to measure tensions down to 20 g. per sq. cm. (in concrete) and to record a dissonance of 0.02 hertz giving a 1-mm. deviation of the spot of light.

It will be clear from what has been said above that a sounding wire gives us a very simple, accessible and accurate means of measurement, which can be applied to an extraordinary number of problems.

Acknowledgment.—The numerous successful constructions, described in the present review of measuring instruments, were carried out with the able assistance of the author's talented colleagues in both Institutes, Engineer D. Golovacheff, Engineer-physicist M. Yakutovich, Physicist I. Polevoi and Physicist V. Lazareff.

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DISCUSSION

MR. L. B. TUCKERMAN[1] (*by letter*).—Anyone engaged in studying the deformations of structures under load will welcome Mr. Davidenkoff's description of the many successful applications he has made of the acoustic strain gage, as a source of many fruitful ideas in testing technique. The success of work of this kind under difficult working conditions depends very greatly upon the skill and ingenuity of the experimenter in modifying his basic measuring apparatus so that it will be reliable under all conditions, and in devising special clamps and other attachments to fit it to the special structure to be studied.

I was particularly interested in his ingenious application of flexure rods (Fig. 8) to the problem of securing accurate parallel motion without the interference of frictional resistance. Flexure plates and flexure rods have long been commonplace structural elements in seismological instruments. Although they were early introduced in the remarkable testing machines built by the late A. H. Emery they have only recently begun to come into their own in the field of materials and structural testing. Testing technique would be much improved if they were more widely used.

The acoustic strain gage affords another illustration of an old idea being rediscovered when independent technical developments make it practical. I do not know who first used the pitch of a stretched wire to measure the deformation of a structure but I have in my files a reference to its use by Pietro Cardani[2] as early as 1888. Our modern vacuum tube technique of acoustic measurements was not then in existence so that Cardani resorted to stroboscopic determination of the frequency of the plucked string. Obviously the method was not at that time ready for extended use.

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[2] Pietro Cardani, "Metodo acustico per la misura di piccoli allungamenti, e determinazione dei moduli di elasticita," *Atti*, Reale Accademia dei Lincei, Series 4, Vol. 5, pp. 892–899 (1889).

CHARACTERISTICS OF THE HUGGENBERGER TENSOMETER

By R. W. Vose[1]

Synopsis

The paper describes the behavior of the Huggenberger Tensometer as determined by the use of a specially designed interferometer calibrator.

It is first shown that under any fixed condition of use the readings of the instrument are proportional to the motion involved, and second that this factor of proportionality varies markedly with different conditions of use. The conditions covered include variations in mounting pressure, material of the specimen, balance and position of the instrument, seating on the specimen, and friction within the instrument. An attempt is made to analyze the causes of these effects and to suggest means for their remedy.

From figures obtained in calibration, the accuracy of the instrument is calculated and the deviations to be expected in routine use are indicated.

Introduction

In the Testing Materials Laboratory of the Massachusetts Institute of Technology a number of Huggenberger Tensometers have been used in strain measurement problems because of their lightness, short gage length, and general versatility. After considerable use, and perhaps abuse, the accuracy of these Tensometers came to be questioned on the basis of wear and lack of adjustment. Calibrations being in order, the author, with the aid of the staff of the laboratory, developed the interferometer calibrator described in the appendix to this paper,[2] and as a sequel to simple calibration investigated the general behavior and accuracy of the Tensometer. It is hoped that the results of this latter investigation will give other users of these instruments a better understanding of their peculiarities and confidence in their indications.

Method:

By calibrating typical instruments under various conditions such as occur in practice the corresponding variations in the indications of the instrument have been segregated and their values plotted or tabulated. It is not intended that results quoted for a given instrument should be applied quantitatively to any other instrument, but rather that the qualitative results should guide the user of another instrument in his manipulative technique and in the interpretation of his results.

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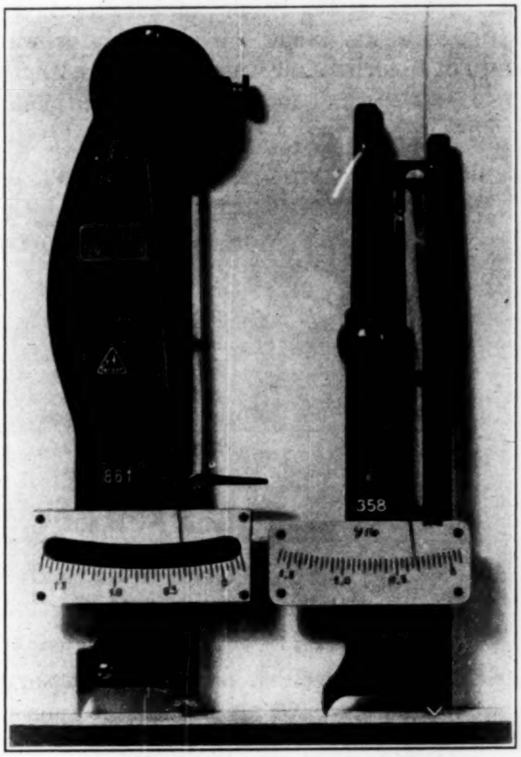
[1] Assistant in Materials Testing, Massachusetts Institute of Technology, Cambridge, Mass.

[2] See p. 873.

Instruments:

Two types of the Huggenberger Tensometer are shown in Fig. 1. Type A is the more sensitive, having knife edge pivots at the principal joints and a magnification of motion in the vicinity of 1200. Most of the investigations described were carried out on this type since the variations discovered are of significance only in the more precise work. The important variations were checked, however, for the type B instrument. The principle of this instrument is the same as that of type A, but the construction is less delicate and the magnification is approximately 1000.



Type A Type B

FIG. 1.—Huggenberger Tensometers.

Behavior of the Tensometer

Linear Relation of Readings:

Owing to reports that the readings of the Tensometer were not proportional to the motion of the tilting knife edge of the instrument, or in other words that the magnification was not constant throughout the range, experimental investigations of the linearity were made, and a mathematical analysis of the lever system was attempted.

In Table I are given the results of a calibration of a typical instrument throughout its range. The calibrator was set to the nearest millionth of an inch at intervals of five wave lengths of mercury green light, or 0.00010750 in., and the type A Tensometer read with a watchmaker's loupe to better than one-tenth of its smallest division, representing approximately 0.000004 in. Three runs were made in this manner and the average of the three tabulated against calibrator settings in the first two columns of the table. From these readings the best average straight-line relationship was calculated by the method of least squares, giving the figures in the third column, and the deviations of the instrument from this theoretical line determined as shown in the fourth column. While these deviations show some system in their variation (indicating a curvilinear relation) their average magnitude is perhaps a millionth of an inch, or an entirely negligible quantity, so that for practical work the linear relation may be taken as accurate.

Table I.—Linear Relationship.

Type A Huggenberger, No. 861. Calibration on cold rolled steel, 16-lb. pressure. Calibrator settings at 0.00010750 in. Average of three runs.

Setting	Reading, 1/120,000 in.	Least Squares Line	Deviation, 1/120,000 in.
No. 1	−14.7	−14.62	−0.08
No. 2	−2.1	−1.78	−0.32
No. 3	11.1	11.04	+0.06
No. 4	24.0	23.88	+0.12
No. 5	36.8	36.71	+0.09
No. 6	49.7	49.55	+0.15
No. 7	62.3	62.38	−0.08
No. 8	75.3	75.22	+0.08
No. 9	88.1	88.05	+0.05
No. 10	101.2	100.89	+0.31
No. 11	113.7	113.72	−0.02
No. 12	126.7	126.56	+0.14
No. 13	139.3	139.39	−0.09
No. 14	152.1	152.23	−0.13
No. 15	164.9	165.06	−0.16

A strict mathematical analysis of the linkage of the Tensometer leads to expressions too complicated to solve, so that the theoretical deviations from linearity due to angularity of the levers can only be approximated. Since the pointer swings through a much larger angle than any other link in the system its angularity will have the greatest effect, and if its half scale swing be taken as 10 deg. the resulting error at the end of the motion from mid-scale will be approximately $\frac{1}{2}$ per cent—arc 10 deg. − sin 10 deg. The ordinary calibration is taken from end to end of the scale, so that if these end points be taken as correct any intermediate point will deviate from the straight line between them by about half this error, or a matter of 0.000002 in., which again is less than any possibility of reading the instrument in use.

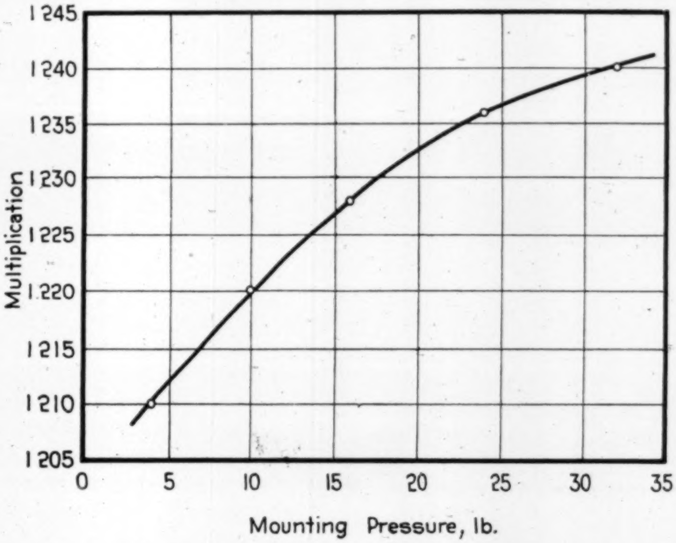


FIG. 2.—Effect of Mounting Pressure. Type A Tensometer (No. 179).

No attempt has been made to carry readings over more than one traverse of the scale by resetting the pointer, but numerous trials have been made without regard to the position of the pointer and a considerable range covered in this way unintentionally. Since the results of these trials agree very closely it would appear that the linear relationship holds through any reasonable number of resettings.

Effect of Mounting Pressure:

The multiplication of the Tensometer varies considerably with respect to the pressure used to mount it on the specimen, as is shown graphically in Fig. 2 for a type A instrument. Part of this effect is due to the depth of seating of the tilting knife edge in the specimen, but the greater part is due to the deflection of the pivot knife edges of the primary lever. Figure 3 shows these knife edges, and it will be seen that they overhang as cantilever beams so that on receiving the clamping load their deflection results in a shortening of the distance between them and the knife edge which seats on the specimen, with consequent change in the lever ratio.

The range shown in Fig. 2 is not extreme, as mounting pressures of upward of 50 lb. may easily be applied with a small knurled head screw. At low pressures the instrument tends to jar loose if any vibration of the specimen occurs, while at higher pressures the effects of friction and also of the hardness of the specimen (to be described later) are much magnified, so that a value of 16 lb. on the instrument has been adopted for ordinary work on metals, and 6 lb. for celluloid, etc. To measure this mounting pressure directly would require in most cases a load indicator incorporated in the mounting device, but a much simpler method is to measure the force required to lift the tilting knife edge just clear of the specimen against the spring of the mounting. In some cases a spring balance or appropriate weights may be hooked to the main lever and the instrument pulled directly

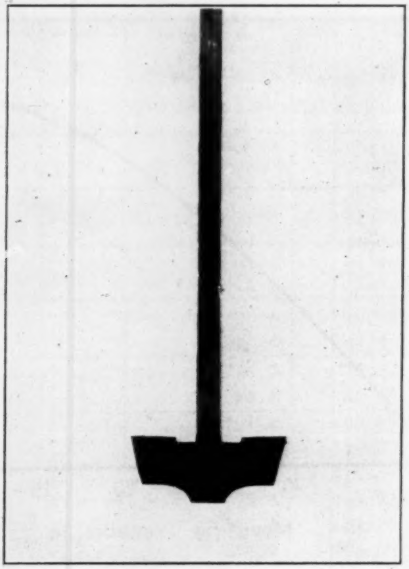


Fig. 3.—Primary Lever. Type A Tensometer.

away from the specimen; or the frame of the instrument may be used as a lever with the fixed knife edge as the pivot, and the force applied as indicated in Fig. 4. This general method is dependent on having a mounting device of considerable springiness, but this is a necessary feature of a good Huggenberger set-up, since any binding due to a stiff mounting will result in a variable pressure and consequent irregularities in the indications of the instrument.

The magnitude of this variation indicates that attention must be given to it in all work, and particularly in cases where several runs or instruments are to be averaged so that the results may be relied upon to better than one per cent. The original calibration should be made at the pressure at which the instrument is ordinarily to be used, and at any other pressure the change in the multiplication may be allowed for approximately from the curve given in Fig. 2. While no other type A instrument has shown exactly the same relation as No. 179, for which the curve is drawn, the general nature has been the same and the numerical differences of minor importance.

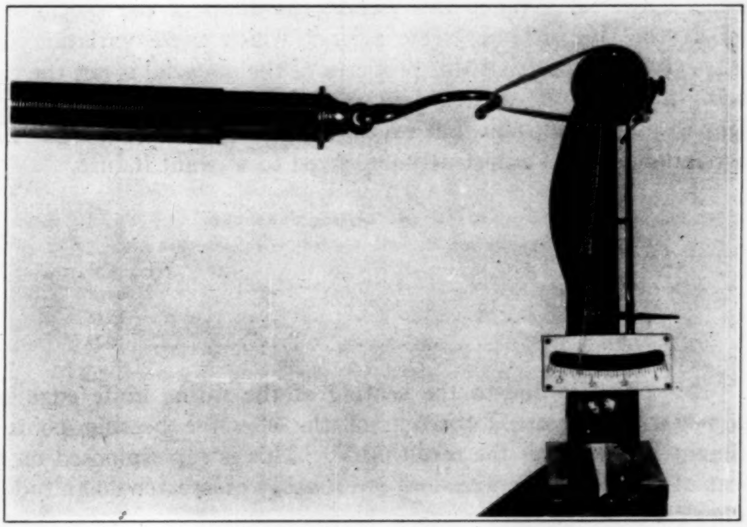


FIG. 4.—Mounting Pressure Determination.

For the type B instrument this variation is not so great as for the type A, as is shown in Table II for two particular mounting pressures. A more rugged construction probably accounts for this better behavior, but attention should nevertheless be given to mounting, and especially to the springiness of the mounting device.

It will be noted in Fig. 6 that the Tensometer shown is mounted with its fixed knife edge on the movable block of the calibrator so that the instrument as a whole partakes of the motion involved. The length and spring of the clamp, which reaches entirely around the calibrator, allow for this relative motion, and it has been found immaterial whether the instrument be mounted as shown or in the reverse position.

Material and Hardness of Specimen:

Figure 5 shows the variations in the multiplication of the Tensometer which were obtained by seating the tilting knife edge on various materials. While the Vickers[1] hardness of the specimen is used as the (logarithmic) scale against which these variations are plotted, it is obvious that this property of the material is not the controlling function. It was thought that the Herbert "flow hardness" might be more significant, but except for giving celluloid a high value the relation was not sufficiently improved to warrant its use.

TABLE II.—MOUNTING PRESSURE.

Type B Huggenberger, No. 358. Calibration on cold rolled steel.

MOUNTING PRESSURE, LB.	MULTIPLICATION RATIO
16	1018
40	1028

This effect is due to the seating of the tilting knife edge into the material and the formation of the effective bearing point at different locations on the resulting V. This is superimposed on the effect of mounting pressure and is probably of greater magnitude at higher pressures.

The magnitude of this variation is not sufficient to require special attention in use except for such unusual materials as celluloid and very hard steel, in which cases the instrument should preferably be calibrated on the material in question, although an estimate of the change in multiplication might be made from Fig. 5.

Balance of Working Parts:

During the course of the investigations the pointer of the type A instrument, No. 861, was replaced by a wire slightly larger than the original, and it was immediately observed that the multiplication of the instrument changed from 1196 to 1179.

A consideration of the effect of an unbalance in the pointer, as was brought about by the use of the larger wire, shows that when the instrument is upright this unbalance exerts a force on the linkage approximately proportional to the displacement of the pointer from the vertical position. Since the linkage and frame of the instrument must distort elastically to this force, the result will be a secondary motion of the pointer proportional to its primary motion. Mathematically, this results in a multiplication ratio different from that given by a simple consideration of the lengths of the levers involved.

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[1] Vickers hardness = Brinell hardness, approximately.

Subsequently instrument No. 861 was equipped with a lighter pointer which was brought to balance by applications of sealing wax. This procedure resulted in a multiplication ratio of 1197 in the erect position, as compared with the original figure of 1196.

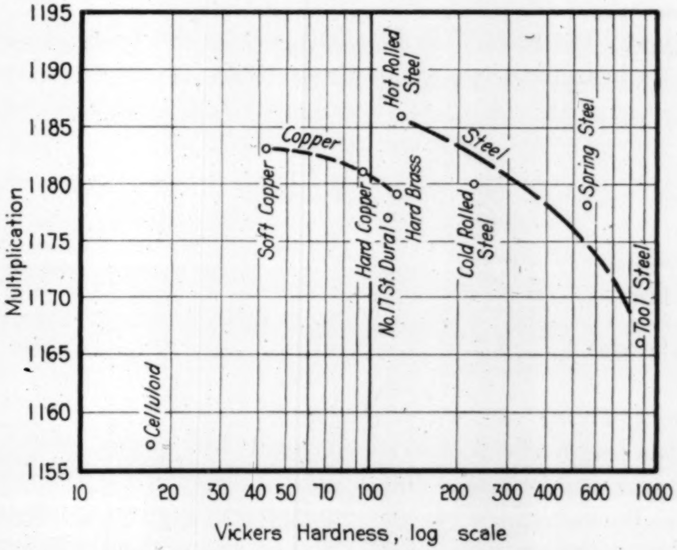

FIG. 5.—Effect of Hardness of Specimen. Type A Huggenberger (No. 861).

Position of Instrument:

When an unbalanced instrument, as instrument No. 861 above, previous to balancing, is used with its plane horizontal the unbalance should have no effect, and the multiplication should be the same as that of the instrument with a balanced pointer. This was borne out for instrument No. 861, which gave a multiplication of 1199 when calibrated with its plane horizontal, as compared with the figure of 1197 with a balanced pointer, and used erect.

When inverted for use on the underside of a horizontal member the unbalanced instrument should show the opposite error to that in the erect position; the result for instrument No. 861 when inverted was 1221, a close check, as may be seen from Table III.

As used on a vertical specimen the axis of the instrument is horizontal but its plane and gage length are vertical. Instrument No. 861 was not tried in this position due to difficulty in mounting on the calibrator, but since the effect of unbalance is approximately constant in this position, rather than variable as when used erect or inverted, the multiplication should be largely unaffected.

As a final generalization the effect of unbalance may be stated to be proportional to the unbalance and to the sine of the angle which the axis of this unbalance makes with the horizontal plane.

Due to a more rigid construction, the type B Tensometer shows less variation with position, although the pointer is not balanced in this type. Values for one of these instruments are compared with those for instrument No. 861, in Table III.

TABLE III.—POSITION.

Calibration on cold-rolled steel, 16-lb. pressure. Type A Huggenberger, No. 861, 0.002 oz. in. unbalance. Type B Huggenberger, No. 358, standard condition.

	MULTIPLICATION	
POSITION	TYPE A, No. 861	TYPE B, No. 358
Erect	1179	1018
Side	1199	1028
Inverted	1221	1040

Seating of Tilting Knife Edge:

The length of the primary lever of the Huggenberger linkage is dependent upon the depth and manner of seating of the tilting knife edge in the material of the specimen, as has been stated. Repeated working of the knife edge in this seating cuts or works the material until a constant bearing is obtained, and up to this point the readings of the instrument are unreliable, with irregularities of one to five per cent being observed the first time contact is made on a fresh surface.

Although loading the specimen several times or tapping the knife edges into the surface may have the desired effect of eliminating these irregularities, the most satisfactory procedure consists of lifting the fixed knife edge clear of the surface and moving the entire instrument back and forth until the pointer has traversed beyond the desired range of motion several times. After this treatment the first reading has been found to vary from the average of a number of readings by less than one per cent in practically all cases, and when used on an elastic specimen the return to the original reading after release of load has been very satisfactory.

Friction:

Friction in the case of a type A instrument in good order is very small, but with a type B instrument or one in which the joints are dirty or out of adjustment, the friction shows as a lag in response to small reversals of motion and also in slight irregularities in the indications. If this friction is not too great its effect can generally be eliminated by tapping the specimen, or better by the vibration from a small electric motor operating in the vicinity. By this latter means a type B instrument may be made to indicate nearly as closely as the more refined type A.

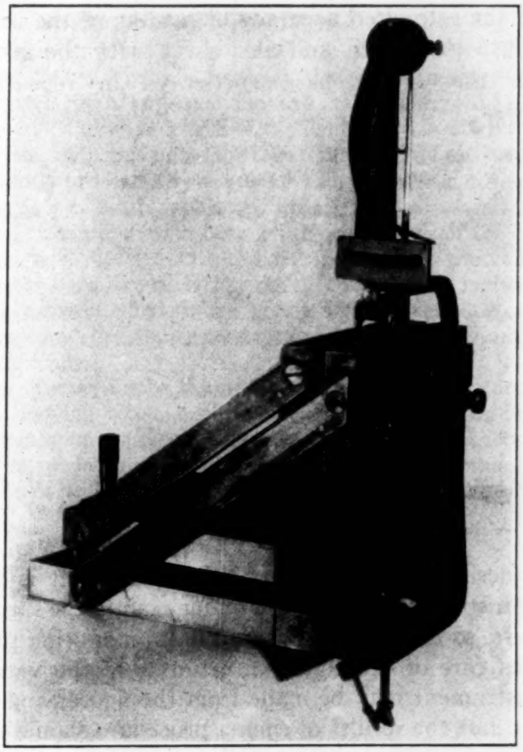

FIG. 6.—Interferometer Calibrator.

Accuracy:

The accuracy of the indications of the Tensometer is dependent on three primary factors, namely, the original calibration, the reliability of the instrument, and the accuracy of visual reading. Experimentally the two latter may be taken together and analyzed on the basis of figures obtained in calibration. A set of readings, taken at random from the many sets obtained, is given in Table IV. From these readings the probable error of a single reading was calculated as 0.2 per cent and the error of the mean of six readings as 0.08 per cent, or 0.000003 in. and 0.000001 in., respectively. These figures are in keeping with the estimated accuracy of reading of the instrument as stated earlier in the paper, and also check with the general run of errors which the author has experienced in repeating various determinations.

TABLE IV.—VARIATION OF READINGS.

Type A Huggenberger, No. 861. Mounted on side. Pressure 16 lb., cold-rolled steel.

RUN	CALIBRATION
No. 1	1204
No. 2	1197
No. 3	1195
No. 4	1195
No. 5	1200
No. 6	1200
Mean	1198.5
Probable error of single reading	2.37 = 0.20%
Probable error of mean	0.97 = 0.08%

If it is desired to utilize fully this accuracy it is necessary to calibrate the instrument in question under exactly the same conditions as those of use, so that the variations due to mounting pressure, etc., may be taken care of exactly. An estimate of the variations for a particular instrument may be made from the figures and plots shown in this paper, but the results of such a procedure should not be relied upon to better than one per cent if the corrections are of appreciable magnitude.

It can be readily seen that an instrument used without regard to the variations outlined above might have the errors due to mounting pressure, material, position, etc., all cumulative, giving a resultant error of five per cent or more. Persistently unreliable results of this sort under unfavorable conditions of use led to the experiments which have been described.

APPENDIX

Calibrator.—The interferometer used for calibrating the Tensometers is shown in Fig. 6. This was specially constructed for this one pupose and therefore has a limited range of motion of 0.005 in. There are, however, provisions for changing the mounting surfaces and the position of the instrument to be calibrated.

The relative motion of the two elements is guided by a pair of short plate knife edges located between the two horizontal plates of the instrument. This forms a parallelogram giving a parallel, though not perfectly rectilinear, motion which is transmitted to the vertical plates on which is mounted the instrument to be calibrated.

To measure the motion these vertical plates carry a pair of optical flats on which interference bands may be observed and counted when illuminated with monochromatic light.[1] For this the green line of the mercury arc (5461Å) as filtered by Wratten No. 62 is used and gives a scale of accepted accuracy reading directly to one-half the wave length of the light, or to 0.000010750 in., and by estimation to one tenth this figure, or the "millionth" of an inch.

The motion is produced by a pair of thin plates laid together to form a beam, which may be bent by the screw visible at the back of the instrument. The relative longitudinal motion between the two plates of the beam resulting from this bending is utilized to move the elements of the calibrator. The proportions of the beam are such that the reduction of the motion of the screw is approximately one hundred to one, giving an extremely fine control without slack or friction, and the ability to set to the nearest "millionth" which can be estimated optically.

Possibility of error in the calibrator arises from lack of parallelism in the motion of the moving block, resulting in different distances being traversed by different points on the block. This has been checked by the interference flats themselves, which are particularly adapted to determining parallelism, and found to be of negligible magnitude although detectable over a long range.

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[1] L. B. Tuckerman, "Optical Strain Gages and Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 602 (1923).

DISCUSSION

MR. F. M. HOWELL[1] (*presented in written form*).—In the Aluminum Research Laboratories we have several tensometers which we have found quite satisfactory for measuring strains under laboratory test conditions. Our calibrations have been made using an extensometer calibrating device described before this Society in 1928.[2] This calibrating device can be read to the nearest 0.000001 in. and provides a ready means of calibrating the instruments on vertical gage lines. For horizontal gage lines a micrometer screw device provides a less sensitive means of calibration.

Our observations on the effects of mounting pressure are in line with Mr. Vose's findings. We have not attempted to measure the effects quantitatively but the tests we have made show variations about as great as Mr. Vose has found.

One point of great importance in the use of these instruments is the necessity for rather frequent calibrations. The knife-edges on the primary lever which Mr. Vose shows in his Fig. 3 have a tendency to become worn, and occasionally chipped, so that there is a change in the lever ratio as well as in the amount of friction. Some indication of the magnitude of the effects of wear is given in a paper presented before this Society.[3]

MR. C. T. SCHWARZE[4] (*presented in written form*).—Mr. Vose has been careful to make all points of the test procedure very clear. There seems to be, however, a slight lack of clarity in explaining the location of the beam of thin plates by means of which motion is imparted to one part of the interferometer calibrator. The idea of attaining micromotion by utilizing relative motion between plates under flexure is excellent and it would be of further interest to have this detail explained.

MR. W. B. KLEMPERER[5] (*presented in written form*).—Mr. Vose, in his paper, refers to mounting pressure. We have found that in our work on light-gage aluminum-alloy structures this pressure has another influence besides the bending of the cantilever knife edges as pointed out by Mr. Vose, namely, the bending imparted to the specimen by the standard type of mounting bracket.

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[1] Engineer of Tests, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Calibration of Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 714 (1928).

[3] R. L. Templin, "Some Factors Affecting Strain Measurements in Tests of Metals, see p. 182.

[4] Professor of Civil Engineering, New York University, New York City.

[5] Manager of Research, Goodyear-Zeppelin Corp., Akron, Ohio.

The standard single Huggenberger mounting bracket applies the mounting pressure to the back of the specimen at one point in the middle of the gage length. This pressure will cause local bending of the specimen and noticeable errors in the strain readings in the low tension stages of a test. We have overcome this difficulty by fitting the brackets with small back bridges of a length equal to the gage length of the tension-meter so that no bending is introduced in the specimen by the clamps.

MR. L. B. TUCKERMAN[1] (*presented in written form*).—Too few detailed studies of the performance of strain-measuring instruments have been published, and Mr. Vose's paper is a welcome addition. In reading it I realized that in our use of strain gages we have paid too little attention to the details of their attachment. It so happens that for other reasons we have always used low clamping forces, fortunately well below the pressures which Mr. Vose found gave significant variations in calibration.

I did not, however, feel so easy in my mind about some work we have been doing with my optical strain gage. This was because even our lightest weight (0.057-lb.) gage required clamping forces on the lozenge of over 5 lb. to hold it securely in place on a propeller blade vibrating at a frequency of over 130 cycles per second. We have, therefore, made a study of the effect of clamping pressure on the calibration of this gage. With this gage clamping forces of over 11 lb. on the lozenge changed the calibration by less than 0.6 per cent on such different materials as duralumin and heat-treated chromium-molybdenum steel. These differences are negligible in comparison with other errors in our propeller investigation, and we would never use such heavy forces in a static test. The smaller change with these optical gages should probably be ascribed to the greater stiffness of the lozenge and the blunter angle at its edge.

I am grateful to Mr. Vose for calling attention to this source of error. It would be very useful if the results of similar investigations of other types of strain-measuring instruments were available for study.

MR. H. F. MOORE.[2]—I should like to make one comment upon the necessity of calibration of extensometers in general. In determining yield strength, or other indicator of elastic strength, it is not at all necessary that the extensometer should read *correctly*, but it is necessary that the reading of the extensometer should be *proportional* to the actual deformation. This is true because in determining yield strength or other index of elastic strength the only object is to determine a stress at which the stress-strain graph deviates slightly from its original path.

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[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

However, if an extensometer is used to determine modulus of elasticity, modulus of elasticity in shear (modulus of rigidity), Poisson's ratio, or the actual strains in a structural member, it *is* vitally important that the extensometer be calibrated.

As a matter of fact, extensometers are rarely calibrated and Mr. Vose has put us in his debt for describing a very ingenious and delicate method for the calibration. The extensometer calibrator depending on a differential thread screw micrometer, described by Mr. R. L. Templin before this Society has been referred to, and I would call attention also to a calibration device using Johansson blocks which is in use in the Materials Testing Laboratory of the University of Illinois.

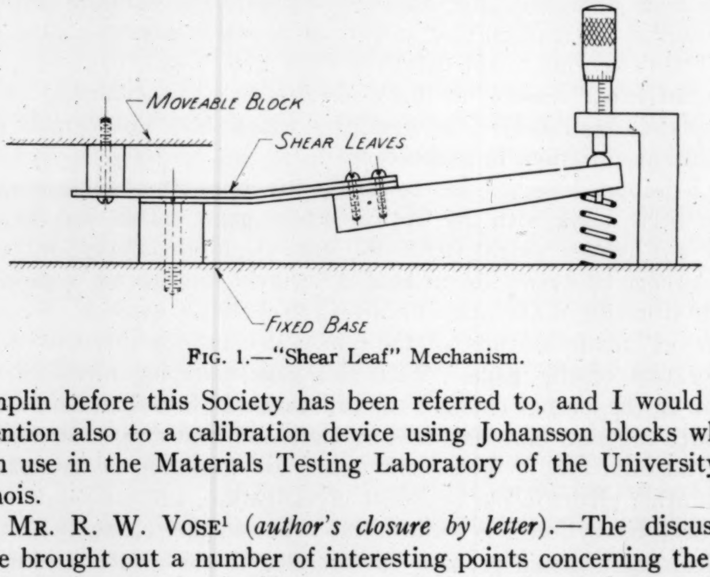

FIG. 1.—"Shear Leaf" Mechanism.

MR. R. W. VOSE[1] (*author's closure by letter*).—The discussers have brought out a number of interesting points concerning the use of the tensometer in specific cases, and the author feels that these points should prove of valuable guidance to other experimenters using the instrument. The remarks of those who have had previous experience in the calibration of this type of strain gage indicate a general agreement with results presented in the paper and form a gratifying corroboration of the work done at the Massachusetts Institute of Technology.

Professor Schwarze's question regarding the mechanism which produces the motion for calibration is best answered by the accompanying, somewhat schematic drawing, Fig. 1, which is self-explanatory.

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[1] Assistant in Materials Testing, Massachusetts Institute of Technology, Cambridge, Mass.

ACCEPTANCE-REJECTION REQUIREMENTS IN SPECIFICATIONS

By H. F. Dodge[1]

Synopsis

Specifications for quality of materials and finished products impose requirements for individual quality characteristics to distinguish between what may be considered satisfactory for a given purpose and what may not. For many characteristics, 100 per cent inspection or testing is not feasible, hence reliance must be placed on sampling a part of the whole. Under these conditions, 100 per cent conformance with requirements cannot be achieved with certainty and errors arising from sampling fluctuations cannot be avoided.

The sampling clauses included in specifications often provide criteria for the acceptance or rejection of lots of a product. These clauses constitute interpretations of the intent of the basic quality requirements and serve as a basis for action. With sampling, certain risks are assumed by both the consumer and the producer. One kind of risk is discussed, and the relationship between (1) the distribution of the risk between producer and consumer. and (2) the choice of acceptance criteria and sample size, is indicated for certain conditions.

This paper discusses briefly the significance of two simple criteria of acceptance and rejection that are sometimes incorporated in specifications, in connection with requirements imposed on individual quality characteristics of a product or material. The discussion relates particularly to characteristics such as tensile strength and weight of coating, where specifications state minimum or maximum requirements that apply to a single unit or test specimen, and where it is feasible to test but a small portion of the whole that may be offered to a consumer for his acceptance.

Specification Requirements:

Any practical discussion of quality and its measurement must deal with variability. Physical and chemical properties of a material vary from spot to spot. The quality of a finished product varies from unit to unit. The quality of any mass of product in respect to any characteristic, such as tensile strength of steel wire strand, weight of galvanized coating of iron sheets, etc., is fundamentally a frequency distribution—not a single fixed value. Examples are indicated in Fig. 1, where the heights of the ordinates represent proportional frequency of occurrence of values of the variable, X, obtained for single units or test specimens.

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Specifications provide requirements for individual quality characteristics to distinguish between what may be considered satisfactory for a given service and what may not. If the specifications require that the diameter of each bolt of a given type shall be within stated maximum and minimum limits, the requirement can be enforced by measuring or gaging each bolt and sorting out those that are nonconforming in diameter. But for a characteristic, such as breaking strength, it is not feasible practically to determine the conformance of each bolt with a specified limit of say 12,000 lb. minimum, because of the damaging nature of the test.[1] This situation exists quite generally in testing physical properties of materials wherever quality can be determined only by destructive tests on specified test specimens. Even for material testing that is non-destructive, there may be no natural discrete pieces which can be separately tested for conformance.[2] Under these conditions, there is no alternative to

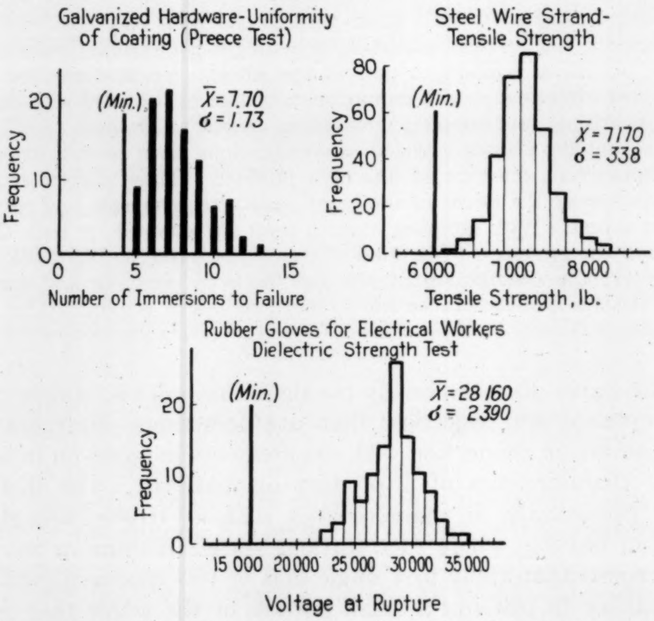

FIG. 1.—Typical Frequency Distributions for Individual Quality Characteristics.

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[1] Ordinarily, the elastic limit is exceeded and the test bolt is permanently deformed even though the applied stress is no greater than the minimum specified.

[2] For such characteristics, a high degree of assurance that the quality desired is attained, can be secured through a program of "quality control" in manufacture. See W. A. Shewhart, "Economic Control of Quality of Manufactured Product," Chapter XXV, D. Van Nostrand Co., Inc., New York City (1931). See also John Gaillard, "Industrial Standardization Especially in the Mechanical Field," pp. 63–66, September, 1933 (printed for private circulation).

testing a relatively small sample of the whole, and the quality of all the parts making up the whole cannot be ascertained. Instead of attempting to classify pieces as conforming or non-conforming, we have the more difficult task of classifying lots or quantities of product as conforming or non-conforming, centering our attention on the *distribution* of quality for families of pieces or for batches of material.

First of all, there is needed a decision as to what distributions of quality are to be considered satisfactory and what are to be considered unsatisfactory. What is the intent of a requirement that specifies merely a minimum limit for tensile strength? Does this mean that, in any accepted lot, no individual piece or no possible test specimen shall have an actual tensile strength value that fails to meet the minimum limit[1] by the smallest of margins? Even though it does, this cannot be enforced.

Does it mean that a small percentage may be allowed to fall below the limit specified? If so, what percentage may be allowed in an individual lot? Further, with what degree of assurance should lots having a percentage larger than this allowable value be prevented from passing? These are some of the questions that should be considered by a specification committee.

The specification of maximum and minimum limits may, in some cases, be taken to imply that the desired or *aimed-at* percentage is zero. However, when sampling is practiced, errors of sampling are unavoidable. For this reason, the *allowable* percentage may be determined, to a large extent, by the number of sample tests that are made and the particular acceptance criterion adopted, as will be brought out in the discussion.

Sampling clauses, agreeable both to producer and consumer, are often included in specifications to provide definite criteria for the acceptance or rejection of quantities or lots of product. These clauses constitute practical interpretations of the intent of the basic quality requirements that are specified and may, in effect, become replacements of the latter so far as action is concerned. When sufficiently complete to provide a detailed basis of procedure, they prescribe what rational subgroups of product should be considered as lots for testing purposes, the steps to be taken to secure a representative sample, the number of tests to be made, and the requirements to be met by the numerical results obtained in those tests. The number of tests and the requirements to be met by the sample data are particularly important items, for they regulate the risks of accepting any unsatisfactory material that may be offered or of rejecting any satisfactory material that may be offered.

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[1] Such limits are sometimes specified so as to allow a wide margin of safety below the extreme limit of variation expected for good commercial product, others may provide practically no margin at all; they do not, in general, give a clue as to what distribution of quality is desired.

On the statistical side, the problem involves a consideration of sampling fluctuations.

Sampling Fluctuations:

In consumer sampling of delivered lots, the aim is to obtain a reasonable degree of assurance that the distribution of quality, in respect to each characteristic, is satisfactory for the service for which the product is intended. The physical sample comprises some n units (or test specimens) of the whole. One measurement on each of the n units gives n observed values (observations) of the variable characteristic, X; the set of n observations is spoken of as a *sample*. The n observations are considered as a composite group rather than as so many individuals, and constitute an observed frequency distribution. We may determine their arithmetic mean, $\overline{X}$ (hereinafter referred to as the "average"), their standard deviation, σ, the fraction, p, of the observed values that fall above or below any given value of X, or any other of the functions[1] of a frequency distribution that are useful for condensing the available quantitative information. However, we may expect the values of $\overline{X}$, σ, and p for the sample to differ from the "true" values of $\overline{X}'$, σ', and p' for the distribution of the product sampled.[2] Barring "constant" errors, either instrumental or personal, the differences between the results obtained by testing samples of the whole and by testing the whole may be regarded as the "errors" arising from fluctuations of sampling.[3] These fluctuations represent an element of uncertainty in sampling and, under certain conditions approximated in practice, their magnitude is inversely proportional to the sample size.[4] The sample size chosen

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[1] See Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, Sections 16–23, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, pp. 464–469 (1933).

[2] The prime notation, $\overline{X}'$, σ', p', etc., is used to designate the "true" or objective values for the universe sampled, and the unprimed notation, $\overline{X}$, σ, p, etc., is used to designate observed values for samples.

[3] It should be noted that the variability of a group of observed values of any variable characteristic is actually made up of two parts—variability of the material itself, and the errors of measurement. By "errors of measurement" is meant the variability observed in taking repeat observations on the same thing. For purposes of clarity, consideration will not be given here to "errors of measurement." It will be assumed either that these errors are small compared with the variability of the material itself or that the specified requirements apply to observed values for specified tests on specified test specimens.

[4] If random samples of n observations each are drawn from a known universe having a distribution characterized by $\overline{X}'$, σ', and p', the values of $\overline{X}$, σ, and p for the samples will have objective distributions of their own, whose standard deviations will be, respectively,

$$\sigma_{\overline{X}} = \frac{\sigma'}{\sqrt{n}},\ \sigma_\sigma = \frac{\sigma'}{\sqrt{2n}},\ \sigma_p = \sqrt{\frac{p'(1-p')}{n}}.$$

These are used as standard units of measure of the errors of $\overline{X}$, σ, and p arising from fluctuations of sampling. Although these equations relate to sampling from an unlimited universe, they may be used as satisfactory approximations in the sampling of limited quantities or lots of product, provided the sample comprises but a small percentage, say not more than 10 per cent, of the units or of the possible test specimens in the lot. This condition is quite generally met in the testing of materials, and is assumed throughout the paper.

and fluctuations of sampling to be expected are, therefore, factors of importance in testing work where interest centers on the distribution of quality for the product sampled.

Any of the measures, $\bar{X}$, σ, p, etc., or any combination of them may be employed for summing up the essential information contained in the sample data, though some are better suited for this purpose than others.[1] Which should be chosen for use in a criterion that is to serve as a basis for discriminating between lots that should be accepted and those that should not?[2] Here is a choice that must be made every time this problem arises. Should the average, $\bar{X}$, of the sample be chosen? Should the fraction defective,[3] p, be chosen? Or should some combination of two measures be used? Before entering into a discussion of the factors to be considered in making a choice, it is desired to indicate the significance of two simple criteria that are often chosen, one involving $\bar{X}$ alone and the other p alone. In the discussion of them, it will be presumed that the only quantitative information available for making a decision as to the acceptability of a lot is that contained in a sample drawn from the lot.

Use of the Average, $\bar{X}$, Alone, in a Criterion of Acceptance:

First, let us consider the criterion:

> Accept the lot if the average, $\bar{X}$, for a sample of n observations is not less than (or not greater than) some stated value, X_L.

Suppose, for purposes of illustration, that for the quality characteristic, weight of galvanized coating for a particular size of steel strand, a specification committee were to adopt a sampling clause that required tests on five ($n = 5$) 24-in. lengths of wire selected in a specified manner from the lot, and required the average of these five tests to be not less than $X_L = 0.980$ oz. per sq. ft. Say that a lot is to be accepted if the average of the sample is equal to or greater than this minimum value of 0.980 and is to be rejected or considered nonconforming if the average is below this value.

To investigate the protection afforded by this sampling requirement requires an analysis of the expected fluctuations of averages of samples of five observations each, when sampling from product having various values of average, $\bar{X}'$, and of standard deviation, σ'. For any given values of $\bar{X}'$ and σ', the averages for some definite proportion of the samples will be expected to fall above $X_L = 0.980$.

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, Sections 27–39, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, pp. 471–482 (1933).

[2] It should be noted that consideration is given here merely to criteria to be applied to the numerical information obtained for one quality characteristic. Meeting these criteria may be thought to be a necessary condition but not a sufficient condition of acceptance. Qualitative information of an unfavorable character may, of course, provide grounds for rejection, quite aside from the findings from the quantitative data. Or again, inconsistency of the data with the quantitative information for other quality characteristics may provide such grounds.

[3] The term fraction defective, p, in respect to any characteristic, signifies the fraction (of the total number of observations) falling outside the limits specified for that characteristic of a unit or test specimen.

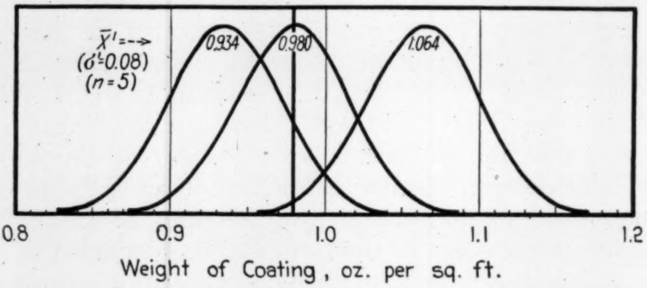

FIG. 2.—Distributions of Average, $\bar{X}$, for Three Universe Values of $\bar{X}'$. (Samples of $n = 5$.)

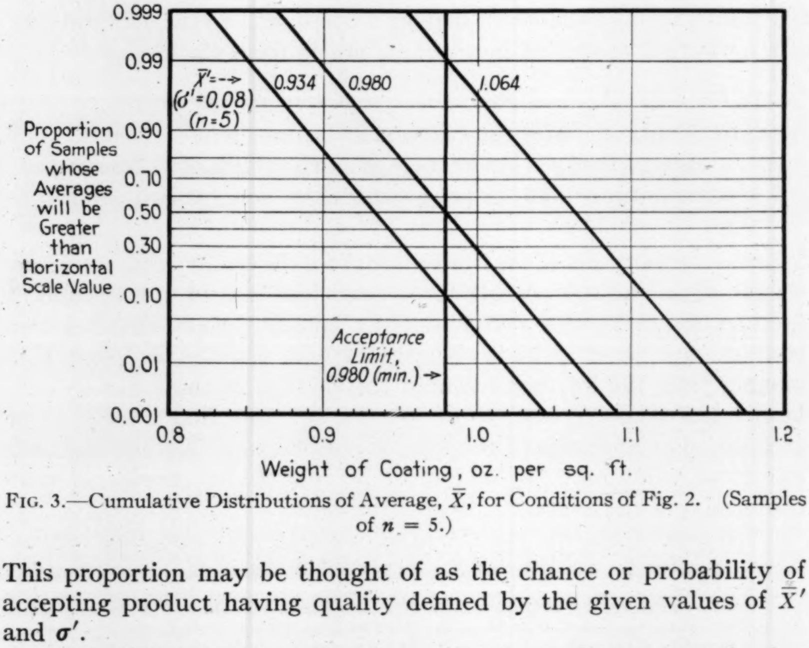

FIG. 3.—Cumulative Distributions of Average, $\bar{X}$, for Conditions of Fig. 2. (Samples of $n = 5$.)

This proportion may be thought of as the chance or probability of accepting product having quality defined by the given values of $\bar{X}'$ and σ'.

We shall determine what these chances are for products having various values of $\bar{X}'$, assuming, for purposes of illustration,[1] a single value of standard deviation, $\sigma' = 0.080$. Figure 2 gives the distribution curves of sample averages for three levels of $\bar{X}'$, and Fig. 3 shows what proportion of the averages may be expected[1] to fall above any given value of $\bar{X}$. From this, it is seen that, under the conditions assumed ($\sigma' = 0.080$ and $n = 5$), for a product having a true average ($\bar{X}'$) of 1.064 oz. per sq. ft., 99 out of 100 samples will be expected to meet the acceptance requirement; while for a product having an average of $\bar{X}' = 0.980$, half of the samples, and for a level of $\bar{X}' = 0.934$, only 1 out of 10, will be expected to conform. If the producer wishes to have a risk of rejection of less than 1 in 100, he would be forced to maintain consistently an average weight of coating of 1.064 oz. per sq. ft. or higher. For a quality level lower than 1.064, his material would often be accepted, but as his average quality fell lower and lower, the chance of acceptance would steadily decrease. The choice of a particular sample size and a particular test limit (0.980 oz. per sq. ft. in this case) has thus determined two levels, one that will, in effect, be considered as a satisfactory level (one for which the *producer's risk* of rejection is very small) and another that

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[1] In practice, σ' would depend on the manufacturing process, and for a given process would probably vary somewhat with the average, $\bar{X}'$, attained.

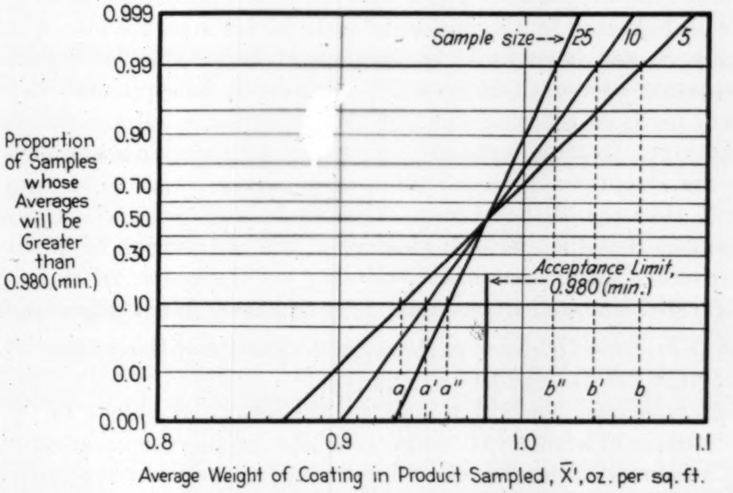

FIG. 4.—Expected Proportion of Samples That Will Meet the Acceptance Limit, "Average = 0.980, min.," for Samples of $n = 5$, 10, and 25. (Standard deviation of universe assumed equal to $\sigma' = 0.080$, throughout.)

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[1] The distribution function for *averages* of samples may, for most practical purposes, be considered normal even though the distribution function of the universe sampled is not normal.

The numerical examples given assume that the universe sampled is homogeneous. A discussion of sampling from a heterogeneous universe, where samples are selected in such a way that each rational sub-portion of the universe is proportionately represented, is given by E. S. Pearson in "Sampling Problems in Industry," a paper read before the Industrial and Agricultural Research Section of the Royal Statistical Society, March 8, 1934.

will, in effect, be considered as a definitely unsatisfactory level by the consumer (one for which the *consumer's risk* of acceptance is small).[1]

The results of setting this sampling requirement are seen somewhat more clearly in Fig. 4 (curve for sample size, $n = 5$). If the producer were to regulate his level by a risk of rejection of, say 1 in 100, and the consumer were to appraise the scheme by the level at which his risk of acceptance is, say 1 in 10, the two significant levels would be 1.064 and 0.934. The narrowness of the spread between such levels, referred to by E. S. Pearson[2] as the producer's and consumer's "safety levels," can be taken as a measure of the efficacy of the sampling plan in discriminating between desirable and undesirable quality. This spread, indicated by the horizontal distance between the lines *a* and *b* in Fig. 4, can be reduced by increasing the sample size, as shown for samples of $n = 10$ and $n = 25$. From this, it is apparent that the safety levels of the producer and the consumer, in respect to *average* quality are, in large measure, determined by the values specified for (1) the acceptance limit for the sample average, and (2) the sample size.[3] The use of the average, $\bar{X}$, alone in a criterion of acceptance presumes that the essential measure of the distribution of quality is its average,[4] and that the variability or dispersion of individual observations around the average is of relatively minor importance.

Use of Fraction Defective, p, Alone, in a Criterion of Acceptance:

Next, let us consider the criterion:

> Accept the lot if none of the measured values for a sample of n observations falls outside the (maximum and/or minimum) limits specified for a single unit or test specimen (that is, if the observed fraction defective, p, = 0).

For convenience, let us say that any measured value that falls outside the limits specified, indicates a "defect." The criterion may then be expressed more simply as:

> Accept the lot if no defects are observed in the sample.

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[1] For the significance of the term "consumer's risk" as here used, see H. F. Dodge and H. G. Romig, "A Method of Sampling Inspection," *Bell System Technical Journal*, October, 1929, p. 613. As applied to the case where acceptance is based on the results of sampling, the term "producer's risk" signifies the probability of failure to accept product of uniform quality resulting from the process in use by the producer.

[2] E. S. Pearson, "Statistical Method in the Control and Standardization of the Quality of Manufactured Products," *Journal*, Royal Statistical Soc., Volume XCVI, Part I (1933).

[3] The value of σ', standard deviation of universe sampled is also a contributing factor; the producer having a high uniformity of quality (lower σ') can safely operate at a lower level of average quality than one whose product shows wide variability.

[4] Methods of determining sample size when the [illegible] is of first importance are given by C. H. Humes, R. F. Passano, and Anson Hayes, "A Study of the Error of Averages and Its Application to Corrosion Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 448 (1930); and by R. W. Crum, "The Number of Specimens or Tests Required for Reasonable Accuracy of the Average," A.S.T.M. Report on Significance of Tests of Concrete and Concrete Materials (separate volume). These methods are based on assumed values of $\bar{X}'$ and σ'.

It is desired to examine the significance of this criterion, particularly from the standpoint of determining how the sample size affects the risks of acceptance and rejection for product of different levels of quality (where quality is expressed simply by the fraction defective, p). Figure 5 indicates the proportion of samples[1] that may be expected to meet the acceptance requirement "no defects in sample," in random sampling of lots for various levels of quality (p') and for sample sizes from $n = 3$ to $n = 50$. For a sample as small as $n = 5$, the producer, to be sure, would have to hold his quality level to 0.2 per cent defective or less if he were to hold his risk of rejection down to 1 in 100 or less. But the consumer would run a 6 in 10 risk of accepting any 10-per-cent defective lot that is submitted to him and a 1 in 10 risk of accepting a 38-per-cent defective lot. This results from the fact that the error of sampling of p is large (relative to p') for small values of n, and is very large indeed when p' is also small. The spread between the "safety" levels (levels of p') of the producer and consumer can be reduced by increasing the sample size, as was true in the case cited above where the average, $\bar{X}$, was used in the criterion. For example, if it were desired to have a consumer's risk of 5 in 10 of accepting a 5-per-cent defective lot, Fig. 5 indicates that a sample size of $n = 14$ would be required, and for a risk of 1 in 10 for a 5-per-cent defective lot, a sample size of about $n = 45$ would be required for the criterion "accept lot if no defects are observed in the sample." It would appear, therefore, that if the consumer wishes to be protected against accepting lots containing a relatively small fraction of defectives, a fairly large sample size is needed when this criterion alone is used. Unless other safeguards are provided, the specification of this criterion and of a small sample may, from one point of view, be regarded as the equivalent of allowing a rather high tolerance for defects in individual lots.

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[1] The distribution of fraction defective, p, (or of the number of defectives, pn) for random samples of size n drawn from a universe having a "true" fraction defective, p', is given by the terms of the Point Binomial:

$$[(1 - p') + p']^n.$$

For example, the probability of occurrence of 0 defects, or of any number, m, of defects, is given by

$$P_0 = (1 - p')^n$$

$$P_m = \frac{\underline{|n}}{\underline{|n - m}\,\underline{|m}} (1 - p')^{n-m} (p')^m$$

See any elementary text on statistical methods, such as G. U. Yule, "An Introduction to the Theory of Statistics," Charles Griffin and Co., Ltd., London (1932). Under certain conditions, particularly when p' is small, say less than 0.10, the Poisson Exponential may be used as a satisfactory approximation to the Point Binomial. Curves, convenient for practical use where this approximation applies, are given by G. A. Campbell, "Probability Curves Showing Poisson's Exponential Summation," *Bell System Technical Journal*, January, 1923, p. 95, and Frances Thorndike, "Applications of Poisson's Probability Summation," *Bell System Technical Journal*, October, 1926, p. 604.

These equations and curves apply as approximations in sampling of finite lots, provided the sample comprises but a small percentage of the lot. See footnote 4, page 380.

Figure 6 gives corresponding information as to the magnitude of the above mentioned kind of risks, when the criterion chosen is "accept lot if not more than one defect is observed in the sample."

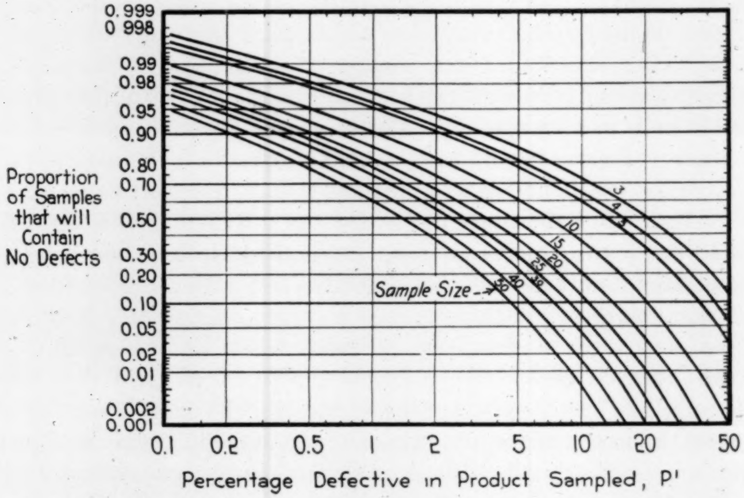

FIG. 5.—Expected Proportion of Samples That Will Meet the Acceptance Limit, "No Defects in Sample."

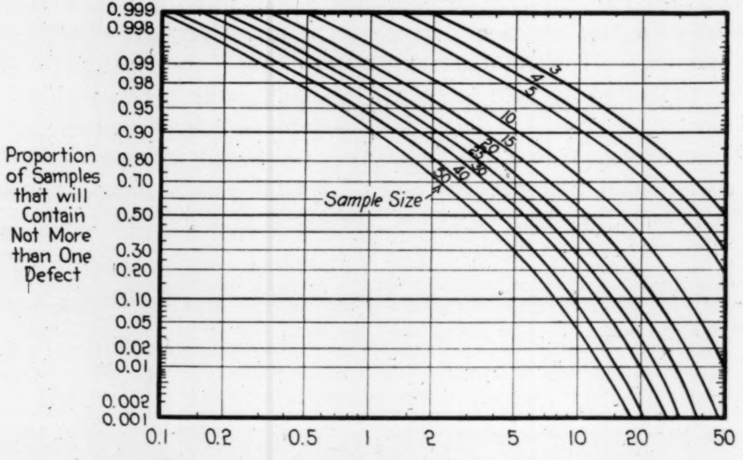

FIG. 6.—Expected Proportion of Samples That Will Meet the Acceptance Limit, "Not More Than One Defect in Sample."

It will be noted that for this criterion, the consumer's risk of accepting moderately defective material is extremely high when small samples are specified.

The use of fraction defective, p, alone in a criterion of acceptance presumes that the essential information contained in a set of n observations can be adequately summarized by merely noting whether any of the individual values falls above or below the specified limit. The proportion of product falling below a minimum limit is frequently a feature of particular interest both to producer and consumer, and the simplicity of this measure is often considered a sufficient justification for its use, at least as *one* of the measures for determining the satisfactoriness of quality. But, generally speaking, the use of p alone is inefficient since it ignores the manner in which the individual observations are distributed along the scale of measurement. Considering the cost of getting data, it would appear desirable that, whenever p is to be used in an acceptance criterion for a variable characteristic, the specification writing committee determine whether some additional measure of quality could advantageously be incorporated in the acceptance requirement. Where this is done, more efficient use of the sample data is assured.

Discussion of Choice of $\overline{X}$, σ, p, etc., for Use in a Criterion of Acceptance:

In making a choice of the one or more measures to be used in an acceptance criterion, a committee responsible for writing specifications should consider such questions as: Which of these measures will be most efficient for distinguishing between satisfactory and unsatisfactory lots? Which are best able to detect those kinds of unsatisfactoriness that are most likely to occur? As has been pointed out elsewhere,[1] if knowledge of the production process indicates that the prevalent cause of trouble is likely to be a bodily shifting of the distribution of quality up and down on the scale of measurement, without appreciable change in the spread of the distribution, the average, $\overline{X}$, would probably be a more sensitive detector of such instances of unsatisfactory quality than either p or σ. If, on the other hand, the most likely cause of trouble is one which will cause a change in the spread of the distribution without appreciable change in the average, the use of $\overline{X}$ alone would be relatively inefficient in detecting unsatisfactory lots arising from this source.

In choosing the one or more measures of a frequency distribution to be used in an acceptance criterion, it would appear essential that a committee take into consideration the magnitude of the sampling fluctuations to be expected, together with the available pertinent information as to the cause and magnitude of normal and abnormal variations in the quality of the product to be sampled. The use of a single measure of quality, such as the average, $\overline{X}$, or the fraction defective, p, may, in some cases, provide all the protection that is needed. However, certain combination measures, such as the average and the standard deviation, the average and the "range,"[1] the average and the fraction defective, the fraction defective and the fraction falling above (or below) some arbitrary limit, etc., may, in general, be expected to provide a sharper discrimination between desirable and undesirable levels of quality than any single measure.[2] Often for a given set of conditions, the size of sample needed for a given protection may be several times as large for one choice as for another.[3]

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[1] W. A. Shewhart, *op. cit.*, Chapter XIX.

Use of Prior Information:

In the foregoing, our discussion of the problem of setting acceptance criteria was carried on with the point of view that the total information available for judging acceptability is contained in the data obtained from a sample of the lot. It is desired to discuss briefly the part played by prior information in consumer testing.

A consumer's or a purchaser's decision to accept a quantity of product should fundamentally be founded on his belief that "the quality of this lot or shipment conforms with the intent of the specified requirements." Such a judgment cannot be certain, but the *degree of belief* that it is rational to entertain in the judgment rests on the character and quantity of the pertinent information that is available to him. As has been pointed out in a previous paper,[1] the sample size needed in any particular instance may be dependent on the past quality history of a product. For example, if the lot under consideration is one of a series which in the past has exhibited "statistical control" at a satisfactory level, and if there is evidence that the conditions of production for the lot were essentially the same as for preceding lots, but little additional quantitative information may be needed to provide the consumer with a high degree of rational belief that current quality is satisfactory.[4] All that he may require of a sample is a sufficient amount of data to test the hypothesis that control has been maintained. A relatively small sample is usually sufficient for this purpose. On the other hand, if no prior information is available, or if prior information indicates unstable or doubtful quality, grounds for such a belief may be lacking and the sample from the lot should be large enough to provide quantitative information which can be considered adequate in itself, by both the producer and the consumer, for estimating the lot's quality and for making a decision as to its satisfactoriness. A somewhat larger sample is usually needed for this purpose.

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[1] A useful "average-range" criterion is discussed briefly by H. F. Dodge, "Statistical Control in Sampling Inspection," presented at a Round-Table Discussion on Acquisition of Good Data, held at the 1932 annual meeting of the Society; published in *American Machinist*, October 26 and November 9, 1932; also available in separate reprint form.

[2] The coefficient of variation (ratio of standard deviation to average), as well as the measure, $(\bar{X} - \bar{X}_a)/\bar{X}$ (where $\bar{X}_a$ = average of the lower two-thirds of the observations) are discussed by E. S. Pearson in connection with the problem of sampling concrete. *Op. cit.*, footnote 2, page 8.

[3] See H. F. Dodge, *op. cit.*, (footnote 1) for a comparison of $\bar{X}$ and p for one set of conditions.

[4] Evidence of "statistical control" over a past period provides a rational basis for the prediction, prior to taking a sample, that a subsequent lot, produced under the same essential conditions, will be comparable in quality with its predecessors.

From these considerations, the question is raised: Would there be an advantage in incorporating in some specifications a double sampling procedure which prescribes two sizes of samples with the following aims: (1) an initial or "control test" sample, relatively small in size, sufficient for carrying out a test for continued control, and (2) a "referee" sample, larger in size, adequate to provide data on the basis of which positive action can be taken as to acceptance or rejection?

If such a sampling clause were written, it would appear that Shewhart's Criterion I for "quality control,"[1] or some equivalent, would be suited for use as an acceptance criterion for the initial sample, and that failure to meet this criterion could be used as a basis for testing an additional sample of such size that the two samples combined would be equal to the "referee" sample prescribed. It would also appear that the criterion of acceptance for the referee sample could be based on a consideration of risks incurred by the producer and the consumer, one kind of which has been discussed in the preceding sections of this paper. With such a plan, statistical control at a satisfactory level should perhaps be considered as a necessary condition, but not as a sufficient condition to test only the smaller initial sample prescribed. By agreement, the consumer should be free at any time to test the larger sample, should he have information of a character that in his opinion justified this step. While it is hazardous to offer a general rule as to the relative magnitude of the two sample sizes in such a plan, it is suggested tentatively that a "referee" sample should be at least three or four times as large as a "control test" sample.

Statistical control of quality in the manufacturing plant has definite economic advantages from the standpoints of both the producer and the consumer, and may be an important factor in determining how much consumer testing is needed in any particular instance. It is possible that some of these advantages could be gained by making use of the principles of quality control in specification writing, in some such way as indicated above.

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[1] W. A. Shewhart, *op. cit.*, pp. 301–318.

Whatever sampling procedure is adopted in a specification, it is important to set forth clearly its aim, and to indicate clearly what agreed-upon action is to be taken when the sampling requirements are not met.

SUMMARY

The selection of a criterion of acceptance or rejection of product requires on the part of a specification-writing committee, among other things, a choice of the quality characteristics for which tests shall be made, a choice of the measure of quality to be used in judging satisfactoriness in respect to each characteristic, and a choice of the risks to be borne by the producer and the consumer when using the criterion as a basis for action. One kind of producer and consumer risk has been discussed, and for a given choice of the magnitude of risks of this kind, a method of determining sample size is indicated when either the average, $\overline{X}$, or the fraction defective, p, is chosen as the measure of quality to be used in an acceptance requirement.

[For Discussion on Statistical Methods, see page 910.—ED.]

APPLICATION OF STATISTICAL METHODS TO THE SOLUTION OF METALLURGICAL PROBLEMS IN THE STEEL PLANT

By W. C. Chancellor[1]

Synopsis

In efforts directed toward the production of uniform steel quality, the desirability of considering plant metallurgical data on a statistical basis is emphasized, since the "exact relationships" governing are complex in their resultant effects and, hence, produce variable rather than "exact" results. A sharp distinction is made between variations resulting from a constant system of chance causes and variations resulting from assignable causes. The application of comparatively recent theory to the detection of assignable causes of variation is discussed and illustrated. Explanations of statistical principles involved are generally of a non-mathematical sort, the object being to stress the practical meaning and usefulness of the mathematical theory. The use of rational sub-groups of data rather than individual values is stressed, since the former can be more accurately treated by present theory. The construction and meaning of statistical control charts, using rational sub-groups of data, are discussed. Typical examples, involving the open-hearth and subsequent processes, are used to illustrate the value of these charts in determining whether statistical control of quality exists or whether variation is affected by the presence of assignable causes. The control chart provides an efficient means of selecting heats or items for investigation in case lack of control is indicated. In many cases assignable causes can be identified and eliminated. This results in a material reduction in the limits of variation, since these are then due to constant chance causes, and statistical control is thus attained. This results in maximum uniformity of quality for the process in question.

Introduction

In the following discussion it is attempted to bring out the usefulness of statistical methods when applied to every-day steel-works problems and to present explanations and illustrations which will emphasize the common sense viewpoint of some of the important principles which many find it difficult to grasp when presented in mathematical terms.

The subject will be treated in a broad, general way and, to a degree, rigor may have been sacrificed for the sake of making certain practical aspects clear. As a further aid to clearness, some of the illustrations and examples used are typical but not actual. Details and refinements are thus avoided in order to emphasize basic ideas. Actual examples are later presented as such to demonstrate the practical value of this method of analysis and attack on plant problems. It is not the intention to present working rules and methods of calculation, as these subjects are adequately treated in the various text books.

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The statistical methods here considered relate primarily to variability and type causes of variability. It is undoubtedly true that *variability* in connection with steel making and *relationship* between steel-making variables constitute major problems in this field. There is a general tendency to deal with averages in speaking of steel-plant problems, and in spite of the fact that the average values are very often satisfactory, the variation over and under the average constitutes the real source of the difficulty. A knowledge of the degree of variability and some conception of the causes of such variability will help materially in defining and classifying steel-works problems.

The steel maker is constantly faced with the problems of better product, greater yield, and economies of process. He alone appreciates the complexities arising from the interrelationships of temperatures, slag and metal composition, deoxidation practices, inclusions, segregation, etc. Granting that continued research may eventually establish fundamental relationships between all the factors involved, it is quite likely that application of this knowledge will be more qualitative than quantitative. Even with such advances in our knowledge, the application of statistical methods of analysis and control to such complex problems will be desirable, for, in practice, these relationships are found to be statistical rather than exact.[1]

If statistics are to be used in this field, the question arises: "Should the statistician be brought into the steel mill or should the steel maker turn statistician?" The former possesses only the tools, the latter knows the peculiarities of the materials and conditions to be worked with, the practical possibilities involved, and the ultimate object desired. As the usefulness of statistics is determined largely by the amount of technical knowledge and common sense brought to bear on the interpretation of the statistics, and as the statistician must rely primarily on his calculations rather than on experience and knowledge of the problem itself, it would seem best for the steel man to make, or at least direct, the application of statistics to his problems, keeping constantly in mind that errors in using the method are as misleading as errors in data or interpretation.

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[1] See W. A. Shewhart, "Economic Control of Quality of Manufactured Product," Chapter IX, D. Van Nostrand Co., Inc., New York City (1931).

VARIATION

The Importance of VARIATION *in Values as Compared with the* AVERAGE *of Such Values.*—It is generally recognized that every quality or item in connection with steel-making problems is subject to variation. While we may state definite or absolute values for such important items as slag analysis, casting temperature, chipping time, etc., in our discussions of these subjects, it is recognized that these values are not absolute and, if such items are of importance, it follows that the variation in these values deserves at least as much attention as the average value. A statement such as: "We like to pour our steel hot or at about 2900 F. (1593 C.)," implies only that the average value is satisfactory. Every steel man knows that in attempting to pour steel at 2900 F. (1593 C.), he will have many heats considerably hotter and considerably colder than this, and if pouring temperatures are important, it at once becomes highly desirable to know (*a*) how much this temperature can vary before results are affected adversely, (*b*) whether or not adverse temperatures are due to assignable causes, or (*c*) whether they represent the normal variation, resulting from a constant system of chance causes, which is to be expected under the conditions of operation.

The value of tolerances or limits of acceptability is generally recognized in connection with manufactured parts and products. The fact that specifications are sometimes unwisely determined does not detract from the value of the principle involved. There is no good reason why similar tolerances or standard limits should not be intelligently placed on intermediate processes and factors of importance in those processes. To return to our example of casting temperatures in the open hearth, if this is an important factor in quality of product, then limits of permissible variation should be set and efforts concentrated on reducing the number of heats outside those limits. Such standard limits are best determined by statistical methods.

The Frequency Curve versus the "Probability" Curve. Variation Due to "Assignable Causes" Versus Variation Due to "Chance Causes." —The value of frequency curves in analyzing steel works data has been pointed out by Daeves.[1] A frequency curve can be made from any sizable group of data, and study of such curves often yields valuable information, but such a frequency curve can be used for the prediction of future results only after it has been established that the variation present is due to several causes, all working *collectively* and *constantly*, and that erratic or assignable causes of variation have been eliminated from the process. Through the use of comparatively recent theory, it is possible to determine whether variation is due only to a "constant system of chance causes" or is influenced by the presence of erratic and detectable causes.[1] The practical importance of this distinction between types of causes of variation will be developed later, also the advantages attained when variation is due only to constant chance causes.

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[1] K. Daeves, "Praktische Grosszahl-Forschung," VDI-Verlag G.m.b.H., Berlin (1933).

A schematic example of the distinction between a frequency curve and a true probability curve, and of the general application of recent theory, is given here to illustrate the major points involved: Suppose a study of the subject has shown that the amount of oxidation (FeO) in open-hearth slags bears a relation to the amount of defects in the finished product. The variation in FeO values then becomes a matter of importance. A frequency curve can be made for the FeO values obtained on each heat in successive monthly periods. Such curves may or may not yield useful and definite information, depending on the degree to which they differ in smoothness, similarity of shape, position and number of heats involved in each. Correct interpretation of such curves is, therefore, quite difficult. If, however, the same data is broken up into small rational sub-groups[2] and these sub-groups used to obtain estimates of the true average value and the true limits of variability,these estimates cang now be used to test for statistical control; that is the absence of assignable causes of variation in FeO. If succeeding periods exhibit this statistical stability, then the frequency curves may be regarded as estimates of a true probability curve, and prediction of future results is justified. If, however, variation in certain periods is found to exceed the limits estimated for constant chance cause variation, it is evident that causes of a detectable and non-constant nature are affecting the results. Under such conditions the frequency curves of FeO are in no sense probability curves, for statistical stability is absent and, hence, they cannot be used for prediction.

The steel maker recognizes many factors which affect, directly or indirectly, the FeO values. If each of these is statistically controlled, the FeO will be also. If the FeO is affected by detectable causes, these may be of two types: (1) Erratic causes which have been allowed to influence results, or (2) to a shift in the balance existing among the constant causes already at work. An example of the first, erratic causes, would be an erratic supply of fuel which would affect the melting rate and, consequently, the oxidation during melting. An example of the second, a shift in balance between the constant system of chance causes of variation, might be a positive change in the proportion of hot metal used in the charge. Erratic conditions are often detected by statistical methods before results are seriously affected. It may happen that lack of control of FeO, for example, will be noted before any noticeable change has taken place in the quality of the product, but if this change in FeO is not given attention and corrected, further changes are likely to occur, and these will be accompanied by serious changes in quality.

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[1] Shewhart, *op. cit.*, Chapter II.

[2] The use of rational sub-groups, rather than individual values, permits a more accurate estimate of the limits of variation due to constant chance causes This will be further developed.

Measures of Variation: Standard Deviation.—If consideration is to be given to the *variation* in a quality as well as to its average value, it becomes necessary to adopt some measure of variation. The usual measures of variation, with their advantages and disadvantages, are discussed in all standard texts.[1] The standard deviation, σ, is generally regarded as the most useful measure of variation and is the only one used in the present discussion.

"CONTROLLED PRODUCT" AND "CONTROLLED VARIABLES"

In the foregoing discussion, the term "control" has frequently been used with respect to certain quality factors and products. Statistical control is referred to, and as this term involves, mathematically, a rather complex conception, it is advisable to attempt a common sense and practical conception of this important term in order that the mathematical aspects, which are fundamental, may be appreciated. Suppose that rejections for steel surface defects are known to be governed chiefly by four factors: namely, iron oxide in the slag, sulfur in the steel, casting temperature, and heating temperature in the soaking pits. Suppose, further, that these four quality factors have frequency distributions of a fixed sort; that is, their frequency curves in successive periods are essentially the same. This is another way of saying that in the different periods the average and the limits of chance variation have fixed values and that none or very few values fall outside of the limits of chance variation.[2]

This variation *within fixed limits*, of the factors responsible for steel defects, implies a variation of rejections for steel defects *within fixed limits*. In other words, so long as the frequency curves of open-hearth and pit temperatures, sulfur in the steel, and iron oxide in the slag are constant, a constant frequency curve for steel rejections can be expected. This constant frequency curve for steel rejections will then have a constant value for the average and fixed limits of chance variation. This means that so long as control is maintained over these four governing factors, the average amount of future surface rejections can be predicted closely and the proportion of the total heats having predetermined amounts of rejections can likewise be predicted. *The product is then said to be statistically stable or under control, statistically.*

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[1] See "Manual on Presentation of Data," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 451 (1933), for a brief but thorough discussion of this subject, also of the probabilities associated with any range of values, $\bar{X} \pm t\sigma$ for the "normal curve" or for data obtained under controlled conditions. The notations used in the present paper are in accord with those of the manual. In the present discussion, limits of chance variation are taken as $\bar{X} \pm 3\sigma$.

[2] In this part of the discussion the objective values of the average and the standard deviation are referred to. The observed values for these statistics are used to make estimates of the true values and should be regarded as such. The accuracy of such estimates will, of course, be affected by errors in the data. In addition, they will fluctuate according to certain statistical laws, this fluctuation depending on the amount of data used in the estimate.

Since the probable combinations of individual values of these four factors in any one heat are limited, and since the frequency with which each probable set of combinations can occur is following some law of chance or probability, the net result, that is, steel rejections, must also follow some fixed law of probability.

For each of the four governing quality factors here assumed, it would be possible to list a large number of secondary factors or causes of variation which, in turn, affect the distribution or frequency of each factor. Even though open-hearth conditions are uniformly maintained, these secondary factors are working constantly to produce variation, say, in the FeO results. A few of these might be mentioned: Variation in composition of pig iron, scrap, fuel, and variation in fuel supply, efficiency of combustion, physical nature of the scrap, speed of melting of the furnace, amount of lime boil, amount of ore fed and charged, temperatures prevailing at various periods of the working of the heat, method of working the heat, etc. If these causes represent a closed system, that is, their effects are limited as is implied by "control," some conception of the meaning of a constant system of chance causes can be obtained. Erratic blast-furnace operation, for example, might result in the pig iron composition becoming a non-constant chance cause of FeO variation in the open-hearth slag. Similarly, a definite change in fuel or an erratic supply of fuel would introduce assignable causes of FeO variation. A similar analysis of secondary factors affecting temperatures, sulfur, and pit conditions might be given. So long as the balance and constant chance limitations are maintained in the multitude of secondary factors, the four assumed primary factors, in governing steel surface rejections, are controlled within limits. In turn, the ultimate objective—steel rejections—is controlled and future results are predictable. The attaining of such a controlled condition results in a marked stability and confidence in operations which is highly desirable as compared with the uncertainty which attends operations in the absence of controlled factors.

CLASSIFICATION OF DATA PRIOR TO STATISTICAL ANALYSIS

Familiarity with Technical and Operating Conditions Is Essential to Proper Classification of Data.—One of the first difficulties encountered in applying statistical methods to plant data concerns the accurate classification of the data to be used. It is in this connection that the plant engineer, using statistics in the solution of his problems, is apt to attain much more satisfactory results than the statistician who can only accept the data presented to him for use, without recourse to the common sense classification and regrouping which the plant man knows from his experience should be made before attempting the statistical analysis. It is much safer to make a too detailed sub-classification of the data than to use at the outset a very broad grouping. In studying open-hearth temperatures, for example, it is obviously wrong to group different types of steel, such as heats made by recarburizing, with those made by catching them on the way down. Since it is known that the speed of pouring ingots and, therefore, the size of ladle nozzle and size of ingot mold are factors in producing ingot cracks, it would be obviously misleading to study the relationship between casting temperature and ingot cracks without segregating these other factors.

The Use of "Rational Sub-groups" of Data.—No specific rules for sampling or grouping of data can be determined without some knowledge of the degree of control existing and the possible or likely causes of erratic variation which may be present.[1] In classifying data or in determining how data for analysis shall be taken, experience and technical knowledge will indicate that there are certain factors which quite probably are "assignable causes of variation." If such a factor is inherent in a known part of the data, then a separate group for analysis should be made for each part. As an example, it may be previously known that FeO and residual manganese are definitely different in rimmed steels and killed steels, even though the steel compositions are generally similar. If this prior information exists, there is no advantage gained in demonstrating it by further analysis.

Since FeO in the slag of a heat of rimming steel is generally known to be an important factor in controlling the rimming quality, it is desirable to identify the maximum number of factors which can be assigned as both favorable and unfavorable influences on the results desired. This leads to "rational sub-grouping" of the data. It is logical to assume, for instance, that composition of the hot metal, size of ladle nozzle, and size of ingot molds may have important effects on results. In an analysis, then, of FeO in rimmed heats, the sub-groups (points on a control chart, which will be discussed later) may be so selected as to contain, in any one group, only heats of a definite type. These types of heats might be those with high silicon in the charge, those poured in a specific kind of mold, those finished by a particular melter, etc. In looking for controllable factors in a problem, the data can be regrouped in various rational ways in order that the suppositions concerning detectable causes of variation may be tested for their correctness.

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[1] The basis for rational sampling is discussed by Shewhart, *op. cit.*, pp. 299, 409–412.

While direct correlation between steel-making variables is highly desirable, it is not essential to marked improvement in results if the principal causes of variation are identified qualitatively and efforts are made to maintain them in the range most favorable to the results desired. The use of rational sub-groups in an analysis is a powerful tool for identification of important factors, and the control chart itself will indicate the degree of success in regulating the controlling factors, once they are identified. In the absence of any other basis for rational sub-grouping, time obviously becomes the best one.

STATISTICAL CONTROL CHARTS, THEIR MEANING AND USE

Purpose of the Control Chart.—The control chart is a graphical means of showing readily whether or not statistical control exists. When control is lacking, those samples out of control are made readily evident by the chart.

It is possibly worth while to attempt a simple and non-technical discussion of these charts and the values and principles involved in them. The following makes no attempt to serve as a working basis for the use of such charts but aims to assist in visualizing their meaning and usefulness.[1]

There are two main types of control charts: (*a*) Charts for average and for standard deviation; (*b*) charts for pieces or percentage rejected on a "go-no-go" basis.[2]

Control charts of type (*a*), that is, charts for average and for standard deviation, consist of three parallel lines, the middle one of which is taken as the expected value of the the average or the standard deviation, depending on which type of chart is under consideration, and the two outer lines represent the limits of variation due to constant chance causes within which the group sample values (points on the control chart) may fall.[1]

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[1] The methods and principles contained in Shewhart, *op. cit.*, are probably the most advanced and most useful application of statistics to manufacturing processes yet developed. For the full development of the control chart, reference should be made to this work.

[2] For discussion of variables and attributes, see Shewhart, *op. cit.*, pp. 40, 41, 71. The calculation of limits and the construction of charts of type (*b*) are less involved than those of type (*a*) and will not be discussed in detail in this paper. Reference should be made to Shewhart, *op. cit.*, pp. 40, 41, 71, 178, 304, 309.

For any specific case, both of these charts depend on the mathematical properties of the fundamental frequency curve of that case, which is based on a large number of single observations, but they differ from the true frequency curve, as generally encountered, in the following respects. The production of a frequency curve of an approximately true theoretical shape requires a large amount of data, although the exact number of observations required depends on the variability of the particular quality being considered. Several hundred observations are generally necessary to determine closely the contour of the frequency curve applying to the case in question. This is especially true of the extremes of the curve which are the parts most affected by erratic conditions and which are of most concern.

The Use of Several Small Groups of Data, Rather than One Large Group.—Decisions on plant problems are frequently required before sufficient data can be accumulated for the construction of characteristic frequency curves. The control charts can be constructed from a much smaller amount of data, and the data can be used more efficiently in the form of a control chart than when treated as a frequency curve of single observations.[2]

A series of random observations of a quality, divided into small *equal* groups of, say, 4, 6, 13, 100, etc., each, provided they come from a single probability distribution, possess certain mathematical relationships to the fundamental frequency distribution, although they may be too few to form a good frequency curve of themselves. It is very desirable to have an understanding of why it is possible to work with these groups of small samples. In the first place, the main object of the analysis, or of the control chart, is to enable us to say whether or not the groups of samples selected come from a large number that are varying according to some probability law or whether they come from a large group which is affected by erratic or uncontrolled conditions.

For simplicity, groups of four rather than any other number which might be chosen will be used in the following discussion. If a few observations, say 48, are available on a certain quality, such as iron oxide in the open-hearth slags, these 48 observations can be grouped into twelve rational sub-groups of four each. If there is no other rational basis for sub-grouping, time can be made the basis.

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[1] When n varies, the control lines will vary accordingly. See Shewhart, *op. cit.*, p. 310.

[2] This follows from the fact that the distribution of the small sub-groups tends to follow the "normal law," while the individual values may follow an unknown law. See Shewhart, *op. cit.*, pp. 211, 267.

For each group of four, the average and the standard deviation can be calculated in the usual way. These averages and standard deviations obtained from small groups will, of course, show considerable fluctuation or differences among themselves, because four, as used in this case, is too small a number to give a "good average."

A better estimate of the true limits of chance variation can be obtained by studying the behavior of the average and standard deviation in twelve groups of four values each, than can be obtained by studying the average and standard deviation estimated from the 48 observations taken as a single group. Suppose for the moment that a great many more than twelve values for the average and standard deviation had been obtained, sufficient to form good frequency curves. We then have a *frequency curve of averages of four* and a *frequency curve for standard deviation of four*. These frequency curves have all the properties of the frequency curve in general. They, therefore, each have a standard deviation.[1] If the standard deviation for *averages* of a given group—four in the present example—is known, then it at once becomes possible to estimate the limits of chance variation for these averages.[2] Any one of these averages which falls within these limits is a "good

TABLE I.—LIMITS OF VARIATION TO BE EXPECTED IN THE AVERAGE AND THE STANDARD DEVIATION WHEN THE NUMBER OF SINGLE OBSERVATIONS USED IN THE CALCULATIONS IS VARIED: THE EFFECT OF "SAMPLE SIZE," n, ON THE VALUES OF THESE STATISTICS, $\bar{X}$ AND σ, FROM A CONSTANT CHANCE DISTRIBUTION.

***Basic Data.*—An "infinite" number of FeO determinations in open-hearth slag have shown an average of 12 per cent and a standard deviation of 3 per cent, and all of these determinations form a "normal" frequency curve.**

Average ± three times the standard deviation = 12 ± 9 = 3 per cent and 21 per cent. 99.74 per cent of all single values will fall inside these limits.

Averages			Standard Deviations		
If n Observations Are Taken for the Average n	The Standard Deviation of Averages Will Be: $\sigma_{\bar{x}} = \frac{3}{\sqrt{n}}$	Hence, 99.74 per cent of the Averages, Based on n Observations Will Fall Within the Limits: $\bar{X} \pm 3\sigma_{\bar{x}}$	If n Observations Are Used to Calculate Standard Deviation: n	The Standard Deviation of the Standard Deviation Will Be: $\sigma_{\sigma} = \frac{3}{\sqrt{2n}}$	Hence, the Standard Deviation, Based on n Observations Each, Will Fall Within the Limits: $\sigma \pm 3\sigma_{\sigma}$ or 3 per cent $\pm 3\sigma_{\sigma}$
1	3.00%	3 - 21%	....	.....	....
4	1.50	7.5 - 16.5	4	1.061	0.00 - 6.18%
9	1.00	9.0 - 15.0	9	0.707	0.88 - 5.12
16	0.75	9.75 - 14.25	16	0.530	1.41 - 4.59
25	0.60	10.20 - 13.80	25	0.424	1.73 - 4.27
64	0.375	10.87 - 13.12	64	0.265	2.20 - 3.80
100	0.300	11.10 - 12.90	100	0.212	2.36 - 3.64
10 000	0.030	11.91 - 12.09	10 000	0.021	2.94 - 3.06
1 000 000	0.003	11.99 - 12.01	1 000 000	0.002	2.99 - 3.01

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[1] This can be calculated in the usual way, but is not generally done, as more satisfactory methods are available.

[2] See Shewhart, *op. cit.*, pp. 184, 185, 212, 312.

average" in the sense that it must be accepted as liable to occur from a constant system of chance causes alone. Conversely, any average which falls outside these limits can be assumed to be the result of erratic causes which warrant investigation. These same generalized remarks apply to the standard deviation.

As it is highly important to have a conception of the behavior of averages and of standard deviations calculated from small groups as opposed to large groups, Table I may be useful in visualizing the situation.

The first part of this table considers the variation in averages expected to occur due to constant chance causes alone, based on the number of samples contained in that average. In this example it is assumed that the true average of FeO content is already known, that is, 12 per cent, and, likewise, that the standard deviation, based on an infinitely large number of samples, is 3 per cent, and that the distribution is normal. If, then, it is attempted to check this average by taking only one observation, it must be accepted beforehand that this observation may fall anywhere between 3 per cent and 21 per cent. If 16 individual slag samples are taken to determine the average, the resulting average can be expected to fall anywhere between 9.75 and 14.25 per cent. Even if ten thousand are taken, the average obtained may fall anywhere between 11.91 and 12.09 per cent.

The point to be noted is that the limits of variation for the average (when n is constant) represent the right and left-hand extremes of the probability curve of averages for the particular value of n, and that any value which falls within these limits need not be questioned for error. Of course, the chances of obtaining values at either of the two extreme limits are rather small, and the probabilities are that the value obtained will lie nearer to the true average than at either extreme. The above illustration is similar to many found in actual practice where successive attempts to determine the average value of an important item give results which differ considerably and which are, therefore, sometimes questioned. If it is known that the quality in question is under control, these differences in averages obtained can be explained, and the averages can be accepted with considerable confidence. In the second part of Table I, the limits of chance variation of the standard deviation are shown for this same basic data.

Control charts of this type cannot be calculated until a fairly satisfactory estimate has been made of the true average and the true standard deviation. Methods for constructing control charts and conditions for their valid use are given elsewhere.[1]

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[1] See Shewhart, *op. cit.*, Chapters XVII–XXI, inclusive.

Discussion of an Actual Control Chart Used in Practice.—Figure 1 shows an actual set of control charts developed for percentage FeO in open-hearth finishing slags of a certain type of steel. Four hundred and sixteen individual heats are involved, these being grouped into 104 groups of four heats each ($n = 4$), selected in chronological order.[1] For identification of individual heats, the different groups are given consecutive numbers and a record kept of the heat numbers in each group. From the data available, the average and the standard deviation of each sample of four were calculated, as well as their limits of chance variation. Control charts were then constructed as shown and the individual groups plotted in chronological order.

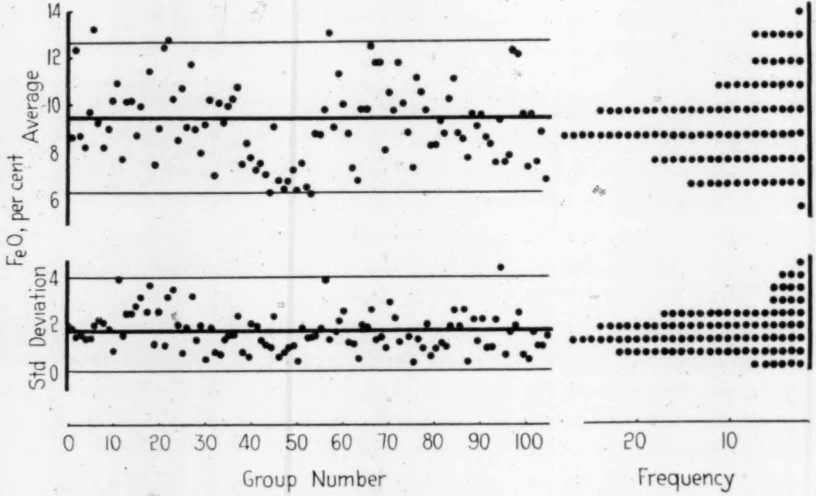

FIG. 1.—Control Chart for FeO in Open-Hearth Slag.

In order to assist in visualizing the meaning of these charts, the individual points plotted in the control charts have been "pushed down" to a base line and grouped in intervals. It is seen that they tend to form frequency curves; that is, we have a frequency arrangement which shows the way in which these averages and standard deviations, based on four slags each, are varying.

By examining the data in Fig. 1, it will be seen that there are a few points out of control, either for average or for standard deviation.

Keeping in mind the errors involved in the estimates of the average and standard deviation, which are necessary for constructing an actual control chart, such as the above, it is now advisable to consider those points on the chart which indicate, in themselves, a lack of control of iron oxide, that is, the presence of assignable causes not affecting the majority of points. In the first place, it will be noted that these points are not greatly outside the control limits, and it is reasonable to suppose that the detection of the trouble causing slight deviations beyond the limits will be more difficult than in cases where the point lies considerably outside the limits.

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[1] There is no other apparent basis for rational sub-grouping.

The histories of the heats involved in points outside the limits were investigated in detail and, in some cases, no definite explanation was found for the variation. In the majority of cases, however, it was found that some factor in the open-hearth charge was responsible for the variation. This refers to the proportion of light and heavy steel scrap, proportion of pig iron and hot metal in the charge, and the composition of the iron itself. In border-line cases, where the points are only slightly outside the limits, this condition may be the result of errors in sampling and in making the estimates necessary for the construction of the chart. However, they deserve investigation, as erratic conditions responsible for excessive variation may be detected, if present, even in such border-line cases.

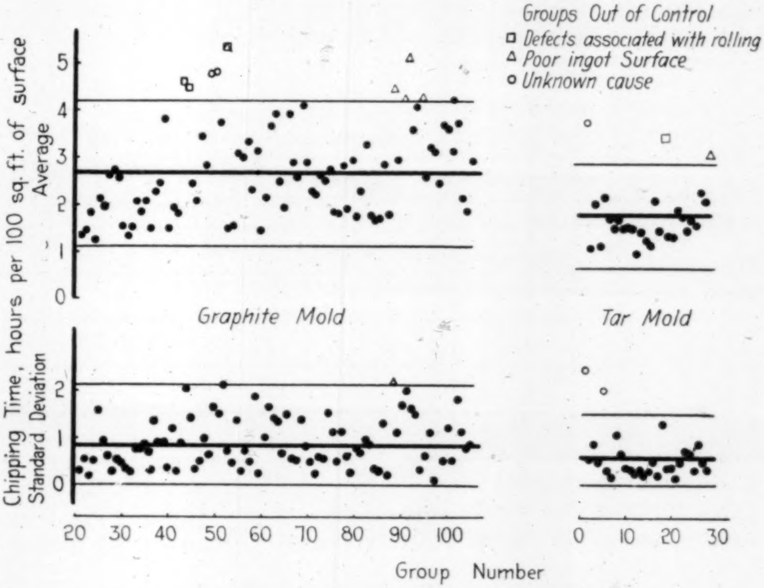

FIG. 2.—Control Chart for Chipping Time.

Additional Practical Examples of Control Charts and Their Use.—In order to study the effect of open-hearth practices on the surface of ingots, blooms, and rounds, so that changes in practice, such as the use of different types of ingot molds and ingot mold coatings, could be measured, control charts were developed for the chipping time per heat in terms of man-hours per unit of surface area.[1]

These charts are shown in Fig. 2. For both kinds of mold coatings, some heats required excessive chipping time.[2] In three groups the trouble was due to rolling mill difficulties rather than to steel-making difficulties, in six groups the trouble was directly due to steel-making details, and for the remaining three groups no explanation was found for the extreme variation. The most interesting feature of these charts is that they show a real difference in both average chipping time and variability in favor of the tar-coated molds.

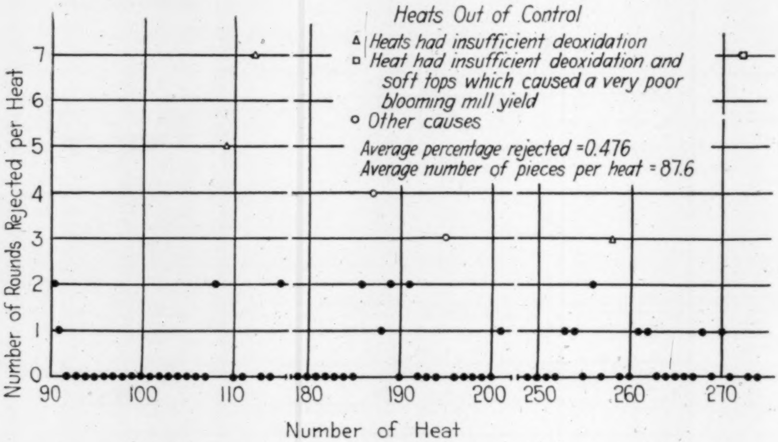

FIG. 3.—Section of Control Chart for Rounds Rejected for Bad Surface.

Figure 3 shows a control chart of type (*b*) for rolled rounds rejected due to very poor surface. The limits of chance variation are not shown in the figure, since they vary with the number of pieces per heat; however, the heats out of control are indicated by different notations. Certain definite indications were obtained by investigating these, the most interesting being those heats lacking complete deoxidation.

It is interesting to note that immediately prior to construction of

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[1] Where the sections vary considerably, surface, rather than weight, is a better unit for measuring chipping time.

[2] Because of lack of space, groups 1 to 20 have been omitted from the charts or graphite-washed **molds.**

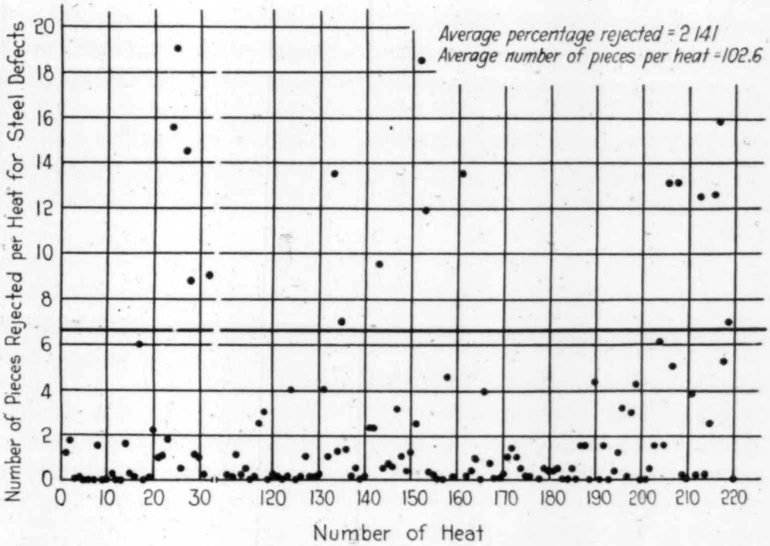

FIG. 4.—Section of Control Chart for Percentage of Rejections Due to Steel Defects, 220 Open-Hearth Heats Made Before an Attempt Was Made to Control Steel Making Variables.

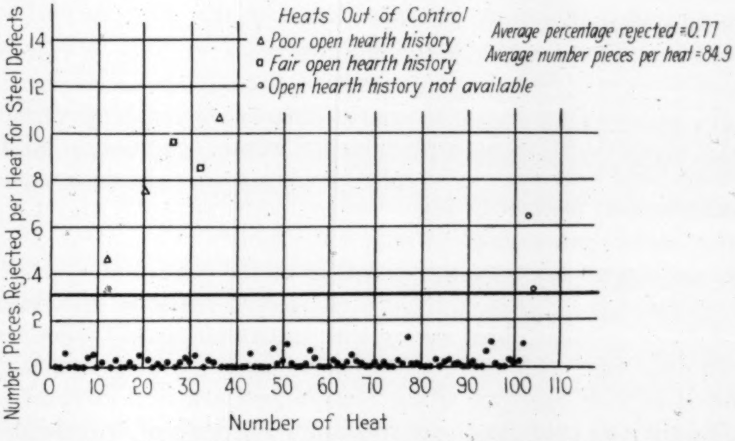

FIG. 5.—Section of Control Chart for Percentage of Rejections Due to Steel Defects After Control Was Established at the Open Hearth.

this chart a very unusual amount of rejections occurred on one heat[1] and the cause of the condition was so evident that an immediate change in amount of deoxidizers was made. The analysis of the chart indications showed that previous heats, lacking sufficient deoxidizers, had resulted in slightly higher rejections than usual, but at the time the connection was not directly apparent. It would appear that had this control chart been placed in effect earlier, this condition would have been detected prior to the occurrence of the glaring case mentioned above.

Several interesting points are involved here. Variation is bound to occur and, without some form of guide, it is difficult to decide on the degree of variation from normal which is worth while investigating; that is, what variation can be left to constant chance causes and what variation is due to erratic conditions deserving attention. The statistical methods which establish limits of variation due to a constant system of chance causes would seem to be the most logical means for distinguishing between those cases which warrant special study and those cases which can be ignored on the assumption that they are inherent in the process. This control chart for rejected rounds, together with control charts for sulfur variation on a certain grade of steel have indicated that the use of such charts tends to detect abnormalities and important changes in conditions prior to the time these changes become noticeable and objectionable in a more obvious way.

Figure 4 shows several sections of a control chart for pieces of finished product rejected per heat on 220 open-hearth heats (rejections here considered were due to a specific types of steel defect.)[2] Twenty-three of the 220 heats had rejections in excess of the chance variation limits. The histories of these heats were carefully examined and, as a result, an effort was made to control certain steel-making variables which appeared to be responsible for the erratic conditions. All but an occasional heat were eventually brought within the desired steel-making control limits. (These occasional heats were given a rating of "poor" before processing.) Figure 5 is a section of a control chart of 104 open-hearth heats made subsequent to the control established at the open-hearth. Seven of these heats showed excessive rejections in the finished product, three having open-hearth ratings of "poor," two, ratings of "fair" and two with no open-hearth record available. In the first run, 10.4 per cent of the heats were out of control, and in the second run this figure had been reduced to 6.7 per cent, but the major improvement effected is shown by the fact that the limit of chance variation had been reduced from seven pieces to three pieces and that the average rejections had been reduced from 2.1 to 0.8 per cent. This is a good example of the narrowing of chance variation limits as well as improvement in the average quality which generally results from the eliminating of "assignable causes of variation."[1]

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[1] This heat is shown on chart as having high loss on blooming mill as well as on inspection.

[2] Only typical sections of the full chart are shown.

Two other examples of the use of control charts are given:

1. After changing from a low percentage of pig iron in the open-hearth charge to a high percentage, the new values for FeO in the slag and residual manganese in the bath were found to be consistently outside of the previously established limits of control. This indicated that a new set of factors was at work. Calculations of control data under the new condition were made and compared with the former conditions as shown below.

	LOW IRON CHARGE, PER CENT	HIGH IRON CHARGE, PER CENT
Average FeO	9.6	11.4
Limits of chance variation, FeO (samples of 4)	6.0 – 13.1	7.1 – 15.7
Average manganese	0.25	0.20
Limits of chance variation, manganese (samples of 4)	0.19 – 0.30	0.13 – 0.26

It is evident from the above that the high iron charge, with its coincident ore charge, has not only raised the FeO and lowered the manganese, but has increased the limits of chance variation in each. A reasonable degree of control was found to exist under both conditions.

2. A definite change in the steel-making practice on a certain type of steel was made and the conditioning time on the bloom surface was observed to note any possible effects. The results are given below:

	METHOD A, HOURS PER TON	METHOD B, HOURS PER TON
Average conditioning time	0.99	0.58
Limits of chance variation (samples of 4)	0.49 – 1.49	0.29 – 0.88

Method B is seen to be better than Method A, both as to average conditioning time and uniformity.

IMPROVEMENT IN PRACTICE AND QUALITY THROUGH (*a*) SECURING CONTROL, (*b*) CHANGE IN PRACTICE OR PROCESSES

Statistical methods are very useful in analyzing a questionable or unsatisfactory manufacturing condition, involving quality, loss of product, etc. The following outline of procedure is suggested for such an investigation:

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[1] The factors responsible for lack of control in Fig. 5 were confined to certain heats and did not have an influence on the other heats.

The quality may be unsatisfactory because (1) the average is unsatisfactory, or (2) variation is excessive, or (3) both average and variation are unsatisfactory. The first step consists in determining whether statistical control exists. If it does not and the presence of detectable causes is thus indicated, these should be sought for and eliminated if possible. Their elimination will greatly affect either the average, variability, or both, and the new (controlled) values for both the average and the limits of chance variation should then be determined. These may, under the controlled conditions, be acceptable. If not, a new *system* of constant chance causes (change in process) must be set up.

Suppose, for instance, the physical properties of the product after heat treatment are the subject of study, and that after statistical control is established, the results are not satisfactory. If the average is in question, it might be raised by a proper shift, say, in chemical composition, and if the latter is under control to the same extent in both ranges, the average, but not the variation, will be affected. If variation is in question, this might be decreased by controlling, within closer limits, either the chemical composition or the heat-treating conditions. New heat-treating facilities, with narrower limits of chance variation, might be the most economical means of securing greater uniformity, particularly if chemical composition is already controlled to the best economical degree.

The formulation of such problems, along the same general lines, but without recourse to statistics, is frequently used. The advantage in using a statistical analysis lies in the more exact formulation of the conditions to be corrected, since detectable and non-detectable causes of variation are differentiated and efforts can be efficiently directed either toward better control of the old process or adoption of a more satisfactory one, as indicated by the results of such statistical analysis.

SUMMARY

Steel-making problems appear to be particularly suited to the application of statistical methods, especially those more recent methods involving the use of small rational sub-groups of data rather than individual values. The justification for a "statistical" rather than an "exact" viewpoint on steel-works problems lies in the complexity of combined causes producing the resulting effects, even though the basic governing laws may be regarded as exact.

Since variation in results must be expected, methods are suggested for determining whether variation is due to a constant system of chance causes alone, or is due partly to detectable or assignable causes. These methods are of much use in identifying this latter type of cause, and the degree of success will depend largely on the rational classification of the data. This condition justifies the application of the methods by the plant engineer rather than by the statistician.

A statistical control chart is used to indicate the average quality, the constant or non-constant nature of the variability present. It also serves as a measure of improvement which may result from elimination of detectable causes of variation and from any change in practice or process which may be indicated as necessary for improvement in case results are not acceptable after control has been established.

In conclusion, it is desired to point out that the application of statistical methods to steel-works problems is in its elementary stages and that further and more general use of statistical methods will help to establish their proper place and usefulness. The foregoing general discussion is given primarily with the hope of arousing further interest, which will, in turn, lead to a broader and more useful application. Two of the principal aims in this discussion are to point out the very great possibilities existing in this field and to offer a non-mathematical viewpoint of some of the important conceptions and details which the present author, not being a statistician, has found difficult to grasp when presented from the statistician's viewpoint.

The author desires to express his appreciation to Dr. W. A. Shewhart and Mr. H. F. Dodge, of the Bell Telephone Laboratories, for their encouraging criticisms and suggestions, and to Mr. J. E. Gould and Mr. H. J. Hand, members of the Metallurgical Department, Lorain Works, National Tube Co., for their helpful assistance in preparing this paper, particularly in the preparation of the data and figures.

DISCUSSION ON STATISTICAL METHODS

MR. D. F. MINER[1] (*presented in written form*).—It is encouraging to note the increased use being made of statistical methods of product analysis, particularly in the metal industries. Simple mathematical interpretations are being given to the former excellent theoretical treatment. These interpretations are necessary to enable manufacturing and engineering personnel to make most effective use of the principles. Manufacturers will be benefited in being able to control more closely the quality of their products, especially those subject to the natural variations of many raw materials, and will be able to recognize lack of manufacturing control and its source. Another benefit which will result from the application of these methods to testing and inspection will be a better situation between supplier and user of materials and products. It is quite probable that many methods of inspection by users have been unfair to the producer and have resulted in unnecessary rejections. On the other hand, it is possible that in some cases more drastic rejections should have been made. A recognition of proper method of sampling and interpretation of test results on these samples will aid in determining a better basis for acceptance and rejection.

MR. E. D. SMITH[2] (*presented in written form*).—The benefits which industry may gain by the application of statistical analysis to the solution of a wide range of problems in production and standardization, as well as in research work, are not fully appreciated. But it appears that the techniques of the method are submerging the true philosophy of this branch of scientific analysis. What should appeal to us most of all is the underlying lesson to be taught—the human basis upon which the statistical analysis is founded. In other words, upon what is the philosophy of your statistical control of industrial manufacturing processes builded?

Mr. Chancellor's paper on statistical methods in a steel plant has indicated that he desires to supply the consumer a product of constant homogeneity. But in doing this there must be a variation. This variation he analyzed with great pains, to distinguish between those of an assignable character and those of a purely random nature. The results indicate the dependence of the quality upon choice and proportion of raw materials, variation in homogeneity of the product, effect of heat temperatures, etc. Clearly, the sound judgment of the technical expert, say for example the engineer, is necessary, as distinct from that of the statistician. Once the limitations of the data are known the presentation of the information in such data is dependent on the man skilled in statistics.

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[1] Manager, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] University Fellow, Department of Industrial Engineering, Columbia University, New York City.

As to the general methods of the quality control of manufactured product, it seems that sometimes the control is not as satisfactory as it would be hoped for. This is often due to faulty analysis of the problem; that is, its data. Quite positive results have been obtained in many cases that, upon further analysis, have been proven to be erroneous.

Much of the present method deals with samples that are assumed to be normally distributed. Attention should be given to the examination of the data for departures from the normal law of distribution. Such procedure requires a rigorous analysis of the process and a specialist in statistics who is also an engineer.

There are a great many problems needing solution in this field. But it seems that the most important thing to bear in mind is the direction in which we are moving and not so much the technique used to get there. The technique is forthcoming once the problem is defined and the functional relationships are known.

The use of the probability analysis of the distributions of individual quality characteristics seems to be the outstanding contribution of the paper by Mr. Dodge. A greater use should be made of such analysis of the data as it removes to a great extent the labor of fitting normal frequency curves to the frequency distributions and then integrating the function.[1] The mechanics of the application of the method needs a simple presentation.

The consumer sets the minimum requirements for the material he is purchasing. Generally there is no premium paid for a product better than the minimum. When this condition exists the producer will aim to manufacture his product so that it will just meet the specifications and be accepted. The average or mean of the test specimens might be used as the criterion. Mr. Dodge has presented another method of determining the acceptance requirements. Samples are assumed drawn from the same population with a unit standard deviation. Written consideration to conditions that depart from this assumption, and normality, are important. By way of example, the relation between tensile strength and Rockwell hardness, as given by Shewhart on page 42 of his book[1] for aluminum die-castings. The results of the analysis of this series of tests, without regard as to how they have been obtained, indicates that there is a stable relation between these two quantities. The tensile strength is irregular from sample to sample, when the 60 tests are grouped into 5 samples of 12 tests each; while the variation in hardness is as might be expected by chance. From the practical viewpoint, however, the results are not satisfactory. Analysis by the methods of this Society would indicate very satisfactory results.

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[1] E. D. Smith, "Dynamical Production Analysis," *Mechanical Engineering*, Vol. 56, No. 6, June, 1934, pp. 331–336.

E. D. Smith, "Control of Production in a Dynamic Economy," *Mechanical Engineering*, Vol. 56, No. 8, August, 1934, pp. 470–476.

There has been shown that the consumer must specify his minimum standard and the percentage of cases below this standard that he will accept. This throws an additional burden upon the producer because he must raise his standards and control the product to within small variations to prevent a probability of his product being rejected; based on a sample to indicate the characteristics of the whole lot. The technique of sampling and control require the use of statistical methods, and it would seem that it must be used much more than it has been employed heretofore. This would of course require that an engineer must be employed who is also statistically minded.

Since the producer is not being paid for the excess quality he is supplying the consumer his effort will be to make a product no better than need be; that is, the mean of the manufactured product, and its deviation about the mean, will be controlled to as close a limit as possible in order to just meet the specifications. But what percentage is the consumer to allow the producer to supply below the minimum? That is, how many items must be above the specification before one below the minimum is to be accepted by chance performances? Is it to be 100 to 1, 160 to 1, 740 to 1, or any other value that one might choose? A knowledge of the fluctuations about the mean, in the form of the standard deviation, will establish these chance relationships. Remembering that the consumer is concerned only with a product to meet his specifications, the distance that the mean of the test sample must be above the acceptance limit would be 2.37, 2.5, or 3 times the standard deviation of the sample to give the foregoing chance ratios. It would seem that the limit of 2.5 standard deviations, or a chance ratio of about 160 to 1, would be quite satisfactory.

The computation of the sample standard deviation, as given by the Society's Committee E-1 in the Manual on Presentation of Data (1933),[1] is

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[1] W. A. Shewhart, "Economic Control of Quality of Manufactured Product," D. Van Nostrand Co., Inc., New York City (1931).

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n} X_i}{n} - \bar{X}^2}$$

which also may be written as

$$\sigma = \sqrt{\frac{\Sigma X^2 - (\Sigma X)\bar{X}}{n}} = \sqrt{\frac{n\Sigma X^2 - (\Sigma X)^2}{n^2}} \quad \text{..........(1)}$$

These equations give the standard deviation of the sample rather than estimates of the universe, hence make no allowance for any "degrees of freedom."[2]

In calculating the standard deviation there is considered only a single variable, the number of degrees of freedom is less by one than the number of observations, that is, $n-1$. When the sum of squares of the deviations, the numerator in Eq. 1, is divided by $n-1$ instead of n the resulting standard deviation is a better estimate of the standard deviation of the population from which the sample has been drawn. Specifically, dividing by $n-1$ overcomes the tendency, inherent in the use of small samples, to underestimate the standard deviation of the population.

Now, facts are obtained to enable one to draw certain inferences; so when one passes from facts to inferences one really fulfills the real and only motive for gathering the facts, and for this reason should make any corrections that cause a bias in small sampling when inferring the structure of a much larger group—a population or universe (so in all cases we should divide by $n-1$).

It is therefore seen that in problems of sampling in *estimating* lot quality dividing by n is to the disadvantage of the consumer because the producer can come closer to the acceptance limit than when allowance for the degrees of freedom which are not made in Eq. 1. Allowing for $n-1$ degrees of freedom the new equations are:

$$\sigma = \sqrt{\frac{\Sigma X^2 - (\Sigma X)\bar{X}}{n - 1}} = \sqrt{\frac{n\Sigma X^2 - (\Sigma X)^2}{n^2 - n}} \quad \text{..................(2)}$$

This adjustment is always made in statistical problems of estimation and inference and should be made here. By way of example, to show the change in the standard deviation between the two methods, take the first nine natural numbers, 1-2-3-4-5-6-7-8-9, and compute the statistic by the two different methods. The mean is $\bar{X} = 5$ and

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[1] Manual on Presentation of Data, sponsored by Committee E-1 on Methods of Testing, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 451 (1933).

[2] Student, "The Probable Error of the Mean," *Biometrika*, Vol. 6, pp. 1–25 (1908).

$$\sigma_1 = \sqrt{\frac{2245-2025}{81}} = 1.65$$

$$\sigma_2 = \sqrt{\frac{2245-2025}{81-9}} = 1.75$$

where the subscripts *1* and *2* correspond to Eqs. 1 and 2 respectively. If the chance limit is about 160 to 1, that is, 99.379 per cent of the cases are above the minimum while only 0.621 per cent will be below, then the difference between the two methods is 0.25 units (= (2.5)(1.75) − (2.5)(1.65)). This indicates that σ_1 is for the sample itself while σ_2 is the best estimate of the universe from which the sample is drawn. The producer gains in the first in that he must produce a product with its mean only 4.125 units above the minimum acceptance limit while in the second, the correct case, the consumer profits by making the producer raise his standards with a mean of 4.375 units above the limit. It is therefore seen that this correction is of importance for estimation and should not be neglected. This is especially shown in the equations. For large values of *n* this correction, quite obviously, assumes less importance.

THE CHAIRMAN (*Mr. Dean Harvey*[1]).—Mr. Smith has called our attention to the fact, which has long been recognized by the Committee on Presentation of Data, that statistical methods are best used by the engineer rather than the statistician, particularly for the reason that data has to be used with judgment and the engineer is the one who knows the circumstances under which the samples were taken, and data secured, so he is in the best position to provide the judgment necessary for the evaluation of the data. This is not any criticism of the statistician, because he is performing a very valuable service in providing this means by which the engineer can judge his data. The engineer however has to learn how to use this new tool, and that is where the statistician can be of great assistance to him in showing him how these methods can be used to advantage in a practical way in determining, in a very definite manner, the properties of a lot of materials.

MR. H. F. DODGE[2] (*by letter*).—With reference to Mr. Smith's comments on the choice of a suitable acceptance criterion, using the average and standard deviation of the sample, I do not believe that all expressed requirements are intended to be equally severe, hence it would not appear desirable to adopt any fixed standard value, such as "2.5 standard deviations" for use in all cases. Experience would indicate the advisability of exercising judgment in making the choice in each particular case, taking into account the needs of the consumer and the capabilities of the production processes. An acceptance requirement of this character that has been found useful in practice was indicated in a recent paper,[1] where the "observed range" for the sample, the difference between the largest and smallest observations, is used in place of the observed standard deviation, as a practical expedient.

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[1] Materials Engineer, Material and Process Engineering Dept., Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

Mr. Smith's comments on corrections for small samples in problems of estimating the parameters of the frequency distribution of a universe from the quantitative data of samples, are in line with the recommendations of our Technical Committee. These corrections have appeared in papers presented before the Society.[2] Referring, however, to the problem of setting acceptance-rejection criteria for use with sampling inspection plans, it is wrong to presume that such criteria of necessity involve *estimation* from a sample. On the contrary, such criteria are most frequently set up to serve as a basis for testing some *a priori* hypothesis and to provide a desired efficiency of discrimination between material of various possible levels of quality, in which case the correction factors referred to are inapplicable.

MR. M. F. SKINKER[3] (*presented in written form*).—The paper by Mr. Dodge gives us mathematical backing for many things that experience has taught us must be true. In the past so much material has been produced without control that specification requirements could not be made scientifically. Since we could not use 100 per cent inspection of such component parts engineers have had to use extremely large factors of safety.

Of course, consumers are interested in getting more uniform products to insure their reliability and to reduce the amount of testing. Many manufacturers do not realize the importance of controlled materials in the building of a large unit or plant. In our company the failure of one item of steel to meet the requirements of a long accepted standard specification delayed for months the completion of two huge units. The interest on the total investment during this period was many times greater than the cost of this one item. When we get component materials under measured control then we will be in a position to avoid delays thus reducing fixed charges during construction and repair.

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[1] H. F. Dodge, "Statistical Control in Sampling Inspection," presented at a Round-Table Discussion on Acquisition of Good Data, held at the 1932 annual meeting of the Society; published in *American Machinist*, October 26, pp. 1085–1088, and November 9, 1932, pp. 1129–1131.

[2] For example, see R. F. Passano, "Controlled Data from an Immersion Test," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 468 (1932).

[3] Assistant Director of Research, Brooklyn Edison Co., Brooklyn, N. Y.

Mr. A. G. Ashcroft.[1]—I have been greatly interested in the application of statistical control in textile manufacturing. While the applications of statistical control in this country seem to be centering mostly around the metal trades, I feel that textile products offer a better and larger field for development because the textile raw materials are much less controlled and offer in themselves large variations before any fabricating is done, causing large variations and dispersions which cannot be measured by the ordinary averages or minimums. With nothing more than averages of a quality measurement of these variable materials one is merely guessing at the result. For the past few years I have, in a modest way, applied statistical criteria. Statistical methods seem to have an excellent field particularly in the acceptance rejection requirements covered by Mr. Dodge. With the use of dispersion or spread in addition to the usual average one has a much better picture upon which to base an acceptance decision. Mr. Chancellor remarked that one of the best features of the use of the criteria is the confidence they give that one is measuring something definite. An average of a quality or a per cent defective does not by itself give that confidence.

I was struck by one of the points made by Mr. Smith that a statistician and an engineer would be required to use the criteria in a practical way. I have found, on the contrary, that the simple criteria outlined in the Manual on Presentation of Data are easily taught to a man of high school education. As a matter of fact the statistical criteria are being applied today in my company by a man of about 40 years of age who had been a foreman but had had no theoretical training of any kind. It took him only a few weeks to understand and apply the mathematics outlined in the Manual and to apply the statistical criteria to not only our own product and research but to the effective evaluation of our purchased materials. I should like to emphasize again the textile field in this connection. I believe an application of the statistical approach is of particular importance to our textile committees. They are today setting up standards and specifications for minimum strengths, weights and regains of yarns, methods of measuring twist and other very definite specifications which, it is my belief, can only be accurately set up if the data upon which the specification rests is statistically controlled.

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[1] Product Engineer, Alexander Smith and Sons Carpet Co., Yonkers, N. Y.

MR. HARVEY.—I think Mr. Ashcroft has done well to point out the fact that these methods are practical for use by ordinary people who have not had the benefit of the higher mathematical training. Many people have come to the conclusion that statistical methods are so difficult that they can hardly be understood by anybody except a statistician. That is not true at all; it is something that the committee has been trying to combat for a long time. I am glad Mr. Ashcroft has brought that out so clearly.

MR. H. J. BALL.[1]—Speaking for Committee D-13 on Textile Materials, we certainly are impressed with the high variability that exists in the results of our tests, and it is readily understood from the nature of the materials with which we work why that is true. We have come to realize the need for better interpretation of data and that the application of statistical methods is the probable answer. I think the work of Committee E-1 along these lines is very timely and I wish to compliment the committee on the clarity of its Manual on Presentation of Data. The material is presented in a clear, concise, and readily understandable form. It is my hope that Committee D-13, as time goes on, will put into its specifications limits and tolerances based upon a better understanding of statistical methods and relationships.

MR. WERNER VON BERGEN.[2]—Similar methods as explained by Mr. Dodge have already been used in grading of wool qualities. Our experience in this respect covers a period of three years.

As most of you may know the quality of wool is based on the average fineness of the individual fiber. This fineness can be measured in several ways. We are using a method which determines the widths of the fiber by projecting the image of the fiber on a white surface where it is measured with a wedge ruler. The minimum fineness of merino wool fibers is 12 microns and it reaches its maximum fineness above 70 microns. The United States standard, consisting of 12 qualities or grades, is based on the diameter.

By measuring 100 fibers of any grade we find that the variation in these 100 fibers can be easily drawn as a curve. Each grade has its characteristic curve. For instance:

In quality 80's, the curve starts with 12 microns and ends with 32 microns, with the peak at about 19.5 microns, while in quality 58's the curve begins with 14 microns and stops at 42 microns with the peak at about 24.8 microns. This shows that with the lowering of the grade the quantity of fine fibers decreases, while the number of coarse fibers increases. Since no fibers coarser than 30 microns appear in the finest wool, a dividing line is placed at that point. The percentage of fibers coarser than 30 microns is in direct relation to the grade. It was found that the various grades contain the following percentages of fibers coarser than 30 microns:

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[1] Professor of Textile Engineering, Lowell Textile Institute, Lowell, Mass.

[2] Chief Chemist, Forstmann Woolen Co., Passaic, N. J.

80's	0–1 per cent
70's	1–3 per cent
64's	3–6 per cent
58's	10–15 per cent, etc.

When the number of fibers coarser than 30 microns increases 20 per cent or more, it is advisable to draw a second dividing line at 40 microns. This is the same procedure as Mr. Dodge applied with his second line.

Mr. Dodge said that for the purpose of control a much smaller number of measurements should be required. This is especially true in the routine control of textile fiber in our plant where we know the qualities required. For an ordinary fiber determination we usually make 100 measurements, for a foreign or outside sample 200 measurements, and to secure a real standard, we measure 500 fibers and in certain cases 1000 fibers.

We trust that this additional information may prove the adaptability of such methods for other industries as well.

MR. DODGE.—In connection with the increasing interest shown in the application of statistical methods to the problems of the Society, it has seemed to some of us that more studied consideration should be given to the sampling inspection and testing rules that are to be used for judging the conformity of quality with the intent of stated requirements. These methods are useful in all sampling problems; they provide a yardstick for measuring the significance of variability in test results and are of assistance in determining how many tests should be taken for any set of conditions. Often the number of tests prescribed is so small that the testing rule appears to be predicated on the continued assumption that the quality submitted is satisfactory, and does not provide adequately for the case where available information, either qualitative or quantitative, casts doubt on such an assumption. This inadequacy tends, in general, to favor the producer. The quality control methods that have been under discussion provide a technique for assisting us in determining when we may feel safe in believing that quality conforms with requirements, prior to taking a sample. If such methods were to be widely used by producers, the need for extensive testing of delivered consignments would be greatly diminished and one of the wants of the ultimate consumer, uniformity of quality, would be better satisfied.

MR. W. A. SHEWHART.[1]—The English committee on the application of statistics in industry, which was formed in 1932 under the auspices of the British Standards Institution, will soon have available for distribution a pamphlet discussing some of the applications being made in connection with their work. I think a copy of this when issued may be obtained from the chairman of the committee, Mr. B. H. Wilsdon, Building Research Station, Garston, Herts, England, or from the British Standards Institution. It is also significant that the Royal Statistical Society, on its one-hundredth anniversary, formed its first section devoted to a particular subject when it established the Industrial and Agricultural Research Section. It is issuing a separate supplement to the *Journal* of the Royal Statistical Society in which papers and discussions given before the section are made available to the public. This annual publication should be of great practical value to those interested in economic standardization and the control of quality of manufactured product. The subscription rate is ten shillings per year and the address is Royal Statistical Society, 9 Adelphi Terrace, London, W.C. 2, England.

MR. ANSON HAYES.[2]—In behalf of the Committee on Interpretation and Presentation of Data, I wish to express appreciation of the two splendid papers by Mr. Dodge and Mr. Chancellor. The purpose of the committee is to encourage the development of the application of statistical methods. The Society is interested first in using statistical methods in connection with research work that is done under its auspices. Second, the methods are of interest to the membership of the Society in assisting in the establishment of closer control of the quality of products that are manufactured and sold. Third, they are of interest in enabling the purchaser of manufactured products to have a better knowledge of the quality of the products they buy. The Committee on Interpretation and Presentation of Data is ever ready to be of assistance in connection with any of these problems.

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